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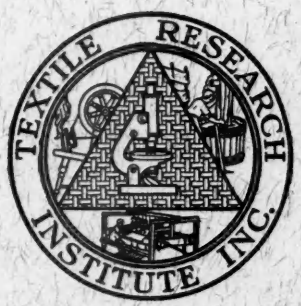

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TEXTILE RESEARCH JOURNAL

VOL. XVII, No. 11 November, 1947

Crystallinity of Cellulose Fibers as Determined by Acid Hydrolysis*

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Abstract

A study of the kinetics of the heterogeneous hydrolysis of cellulose indicates that there are two distinct rates which have been attributed to the rapid hydrolysis of the loose amorphous regions and to the slow hydrolysis of the dense crystalline portions of the fiber. Based on this interpretation, a simple method for the determination of the relative proportions of crystalline and amorphous cellulose has been developed. The method was applied to a series of natural and regenerated cellulose fibers and the resulting "degree of crystallinity," defined as the amount of crystalline cellulose in a sample expressed as a decimal fraction of the total cellulose, was tabulated. Results obtained on four samples of rayon are very well correlated with crystallinity data derived from x-ray diffraction patterns. There is apparently no correlation between the degree of crystallinity and the tenacity of five samples of rayon. In six samples of Stoneville cotton there appeared to be no relationship between the degree of crystallinity and crystallite orientation. There are indications that the ultimate elongation and the moisture absorption of the rayon samples are proportional to the content of amorphous cellulose.

ACCORDING to the present theory, cellulose fibers consist of long-chain molecules which, depending on their arrangement in different parts of the fiber, form highly organized crystalline, as well as more randomly arranged, amorphous regions. Individual cellulose molecules may pass through several crystalline and amorphous areas. The ratio of crystalline to amorphous material is considered to have an important influence on the physical properties of the fiber [15]. Various attempts have been made to determine this ratio. Rather rough estimates have been based on x-ray diffraction patterns [15] and, recently, chemical methods have been developed based on the assumption [10, 11] that chemical reactions proceed much faster in the loose amorphous regions than in the dense crystalline regions. Nickerson [16–19] and Conrad and Scroggie [5] employed the hydrolysis and catalytic oxidation of cellulose for this purpose, whereas Goldfinger, Mark, and Siggia [9] used the oxidation of cellulose with periodic acid. In both cases rate curves were obtained which could be interpreted as a superposition of a rapid reaction in the amorphous areas on a slow reaction in the crystalline areas. A different approach was made by Assaf, Haas, and Purves [1,

* Presented before the Division of Cellulose Chemistry at the spring, 1946, meeting of the American Chemical Society, Atlantic City, New Jersey, and also at the Brooklyn Polytechnic Institute Seminar, March 29, 1947.

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2] by application of the experimentally difficult thallation technique. Applicability of these methods is limited because of their lack of reproducibility and simplicity, and the fact that straightforward mathematical interpretation of the data is not possible.

In the present study an attempt was made to overcome the difficulties mentioned above by the selection of a cellulose reaction which would permit relatively simple experimentation and mathematical interpretation. Such a reaction was found in the heterogeneous hydrolysis of cellulose by acid at elevated temperatures. It has already been indicated by Davidson [6], who did not attempt a quantitative interpretation, that the observed slow-down of the heterogeneous hydrolysis of cotton cellulose caused by prolonged action of acids was due to the more rapid disappearance of the amorphous portions of the sample. Furthermore, it has been found in studies of the kinetics of wood saccharification [13, 14, 20] that the hydrolysis of the more acid-resistant part of the cellulose could be described as a simple pseudo-monomolecular reaction, and that there was also an easily hydrolyzed portion present which hydrolyzed so quickly that its rate could not be determined.

Since the transition from crystalline to amorphous regions is not clearly defined, the terms "crystalline" and "amorphous" are somewhat arbitrary in that they refer to the distinction made by the particular reaction employed. It may well be that the percentage of "crystalline" cellulose found is also dependent on the chemicals used in its determination.

Assuming that there is one specific reaction rate for the amorphous cellulose and another for the crystalline cellulose and that the over-all reactions from insoluble cellulose to glucose (or some other soluble low-molecular-weight polysaccharide) for both the crystalline and the amorphous regions are first order, one may write,

$$r = c + a = c_o e^{-k_c t} + a_o e^{-k_a t}, \qquad (1)$$

where r is the total unhydrolyzed cellulose at time t; c and a are the concentrations of unhydrolyzed crystalline and amorphous cellulose at that time; c_o and a_o are the initial concentrations of the two components; and k_c and k_a are the respective rate constants. Since hydrolysis of amorphous cellulose is assumed to proceed much faster than that of crystalline cellulose, the time will be reached when a becomes negligible with respect to c, the second term of equation (1) vanishes, and the remainder may be written in the form

$$\ln r = \ln c_o - k_c t \cdots c \gg a. \qquad (2)$$

Hence, if the unhydrolyzed residue is measured at various times, and its logarithm plotted against time, a curve should be expected which becomes linear when practically all the amorphous cellulose has disappeared. According to equation (2), extrapolation of the linear part of the curve to zero time should give the logarithm of the initial concentration of crystalline cellulose. If the concentrations are expressed in percentage of the original sample weight, the extrapolated value would give directly the percentage of crystalline cellulose in the sample.

The amount of amorphous cellulose (a_o) in the original sample may be obtained by subtracting the amount of crystalline cellulose from the total cellulose. Or, alternatively, it may be found as the intercept at zero time by plotting against time the differences between total residue and crystalline cellulose on a logarithmic scale after the curve for the crystalline cellulose has been derived.

From the preceding equations, results obtained by the hydrolysis method for crystalline and amorphous cellulose should be independent of the rate of hydrolysis—that is, independent of temperature and acid concentration.

The hydrolysis method described in this paper is based on equations (1) and (2). Acid is permitted to act on a series of small cellulose samples under controlled conditions for predetermined lengths of time, and the unhydrolyzed cellulose is then measured by weighing the hydrolysis residues. These residues also contain insoluble, dark-colored, humic substances, formed from glucose under the conditions of the reaction. Proper allowance for their contribution to the residue weight was made by measuring the rate of formation of humic substance from glucose. Application of the hydrolysis method for the measurement of crystallinity of several cellulosic materials is described in the following paragraphs.

Experimental Procedure

Hydrolysis

As many duplicate 0.2-gram samples as there were hydrolysis periods to be employed were weighed into 25- by 200-mm. test tubes. All the samples for a

complete hydrolysis determination, including additional samples for moisture and cellulose determinations, were weighed out at one time in order to minimize the effect of fluctuations in moisture content. Sample weights were held within 1 mg. of 0.2 g. The early part of the work was done with ordinary test tubes with rubber stoppers. Because the stoppers corrode fairly rapidly, special test tubes having outside-ground, standard-tapered caps with glass hooks and springs were used in the later work. (An inexpensive way of making these tubes is to cut off the bottoms of 25- by 40-mm. weighing bottles with $ No. 29/12 outside-ground covers and join them to test tubes so that an over-all length of about 200 mm. is obtained.) To avoid slow evaporation of the acid, the ground-glass covers were greased with an acid- and heat-resistant silicone stopcock grease. The test tubes were placed in a constant-temperature glycerin bath which for most experiments was kept at 100.0° ± 0.1°C. Standard hydrochloric acid was used as the hydrolyzing agent. In a 2-liter boiling flask, provided with a reflux condenser and a sidearm which penetrated the side wall of the flask about 20 mm. above the bottom and reached inside almost to the bottom, the acid was brought to a boil while the sidearm was closed with a short piece of rubber tubing and a pinch clamp. The flask was located at such a height that acid could be easily withdrawn through the sidearm into the test tubes in the bath. When the acid had reached the boiling point, the test tubes were filled to a permanently engraved mark indicating a volume of 50 ml., and closed as speedily as possible. In this way the acid assumed the temperature of the bath practically from the beginning. After having been in the bath for the predetermined periods of time the tubes were removed and their outsides quickly rinsed with water. The contents were then filtered through dried and previously weighed 15-ml. fritted-glass crucibles of "fine" porosity, washed with distilled water, dried at 103°C, and weighed in tared weighing bottles. The residues filtered fairly quickly provided they were not allowed to suck dry before completion of the washing. The residue-weights were expressed as percentages based on the original sample weights. With normal care on the part of the technician, agreement between duplicate samples was easily obtained within several tenths of a percent.

Erlenmeyer flasks used at the start of this investigation, which were shaken at frequent intervals, were abandoned when it was found that the same results could be obtained in test tubes without shaking.

TABLE I. DESCRIPTION OF CELLULOSE SAMPLES

Material	Description	40%-Angle* (Degrees)
Cotton linters	Purified; fluidity: 10 rhes in 0.5% concentration	—
Raw cotton A	Stoneville 2B	—
Raw cotton B	Wilds 13	—
Raw cotton C	S × P	—
Raw cotton D	Stoneville 5	27.6
Raw cotton E	Stoneville 5	28.3
Raw cotton F	Stoneville 5	30.0
Raw cotton G	Stoneville 5	34.8
Raw cotton H	Stoneville 5	36.4
Raw cotton I	Stoneville 5	36.9
Mercerized cotton B1	Mercerized under maximum tension	—
Mercerized cotton B2	Mercerized without tension	—
Ramie	Only partly degummed	—
Fortisan rayon	High-tenacity saponified acetate	—
Cordura rayon	High-tenacity viscose	—
Fiber G rayon	High-tenacity viscose	—
Textile rayon	Viscose rayon	—
Special rayon	Viscose, spun without appreciable tension	—

* The "40%-angle" is the angle on the 002 x-ray diffraction arc between the point of maximum absorption and a point whose absorption exceeds the minimum absorption by 40 percent of the difference between the maximum and minimum absorption.

Materials

A short description of the cellulose samples which were used in this study is given in Table I. The three samples of raw cottons A, B, and C were in the form of rovings. The six cotton samples D to I were 22s yarns selected from different growth areas and seasons so as to represent a wide range of crystallite orientation as shown by their so-called 40-percent-angles [4], the 40-percent-angle being the angle on the 002 x-ray diffraction arc between the point of maximum absorption and a point whose absorption exceeds the minimum absorption by 40 percent of the difference between the maximum and minimum absorption.

The two samples of mercerized cotton were prepared at this Laboratory by C. F. Goldthwait from 17.5/1 yarn spun from Wilds 13 cotton (cotton B). Both samples were skein-mercerized in 23-percent sodium hydroxide plus 2-percent Dypenol SE (wetting agent) for 4 minutes at room temperature, then washed, soured, washed again, and dried at 210°F.

One sample (cotton B1) was mercerized under exceptionally high tension and dried under tension, which resulted in a total stretch of 3 to 4 percent. The other sample (cotton B2) was treated with caustic under no tension, with a resulting shrinkage of 27 percent.

The ramie was an American-grown sample, raised in a greenhouse, partially degummed with a soap-soda solution, and bleached.

Fortisan is a high-tenacity commercial rayon made by saponification of high-tenacity acetate rayon. The yarn contained 4.3 percent oil as determined by extraction with alcohol. Part of the oil remained in the hydrolysis-residues and was removed by washing the residues with about 10 ml. of alcohol in the fritted-glass crucibles before drying.

The Cordura, Fiber G, and textile rayon used were the regular commercial viscose products with the exception that the finishing treatment with oils or other agents had been omitted. The special viscose fiber was prepared by a commercial firm in a special experiment without any appreciable tension and without the normal purification treatment.

Results

Effect of Liquid-Solid Ratio

Samples of purified cotton linters weighing 0.2 g. were hydrolyzed with 20, 50, 100, and 200 ml. of 6*N* hydrochloric acid at 100°C for 1 hour. The weights of the white residues expressed as percentages of the original sample weights are shown in Table II. Although the liquid-solid ratio was varied from 100:1 to 1,000:1, this had no influence on the weight of the residue, which confirms the monomolecular nature of the reaction. At longer hydrolysis periods, the residues became progressively darker because of the formation of the black, insoluble humic substances previously mentioned. This reaction, as described in the paragraphs which follow, is not monomolecular, so that for longer periods of hydrolysis the somewhat contaminated residues are no longer entirely independent of the liquid-solid ratio. Therefore, a constant liquid-solid ratio of 250:1 was used in this study.

TABLE II. PERCENTAGE OF COTTON LINTERS REMAINING AFTER THE ACTION OF 6*N* HCl AT 100°C FOR 1 HOUR AT VARIOUS LIQUID-SOLID RATIOS

Liquid-solid ratio	Residue based on original sample weight (%)
100:1	80.2
250:1	80.3
500:1	80.4
1,000:1	80.0

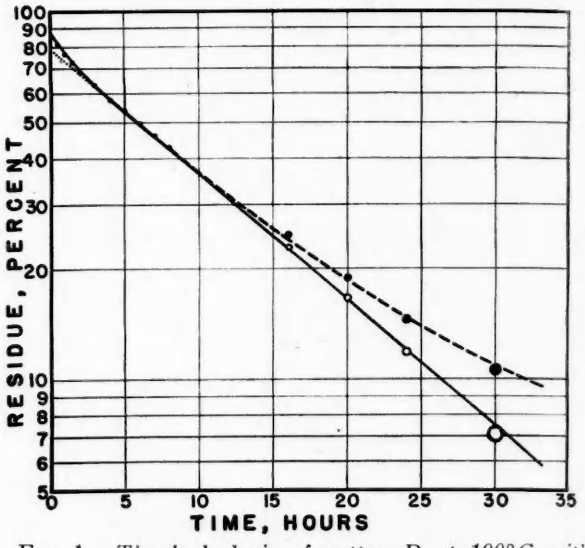

FIG. 1. *The hydrolysis of cotton D at 100°C with 6N hydrochloric acid. The black dots and the broken line represent the observed residues; the circles and the continuous line show the same data after correction for humic substances. The dotted line shows the extrapolation to zero time.*

Influence of Formation of Humic Substances

When samples of cotton D were hydrolyzed with hydrochloric acid for various lengths of time and the percentages of residues obtained were plotted against time on a semilogarithmic scale, the broken curve shown in Figure 1, which is typical of results obtained with other cellulosic fibers, was obtained. The broken curve has the initial curvature according to equation (1) but fails to become linear as

TABLE III. RATE CONSTANTS FOR FORMATION OF HUMIC SUBSTANCES FROM GLUCOSE BY EQUATIONS (3) TO (5)

Normality of HCl	Temperature (°C)	Rate constant: Concentration expressed as g./100 ml. (100 ml./g./hr.)	Rate constant: Concentration expressed as % of 0.2 g./50 ml. (2.5×10^4 ml./g./hr.)
6.0	100	3.5	0.014
4.0	100	1.5	0.006
2.5	100	0.5	0.002
6.0	95	3.0	0.012
4.0	95	1.0	0.004

would be expected from equation (2). Deviation from linearity is accompanied by a progressively darker color of the residues caused by their contamination with insoluble humic substances formed from glucose. Little is known about the constitution of these substances [3] and nothing about the kinetics of their formation. According to Schweizer [22], they are polymerized dehydration products of sugar. In order to measure their contribution to the weight of the hydrolysis residues and to arrive at the net weights of unhydrolyzed cellulose, the rate of humic-substance formation was determined.

Duplicate samples of exactly 0.1, 0.15, and 0.2 g. of glucose were heated with 50-ml. portions of hydrochloric acid for various lengths of time, using the same apparatus and procedure as for cellulose samples. Determinations were made at 95°C and at 100°C with 6*N*, 4*N*, and 2.5*N* acid, to correspond to conditions employed in the hydrolysis measurements on cellulose. As the time of heating was increased, the acid solution turned yellow, then brown, and finally a black amorphous precipitate appeared which was filtered, dried, and weighed. The results with 6*N* and 4*N* hydrochloric acid at 100°C are presented as circles in Figure 2. It can be seen from Figure 2 that the formation of humic substances is not monomolecular.

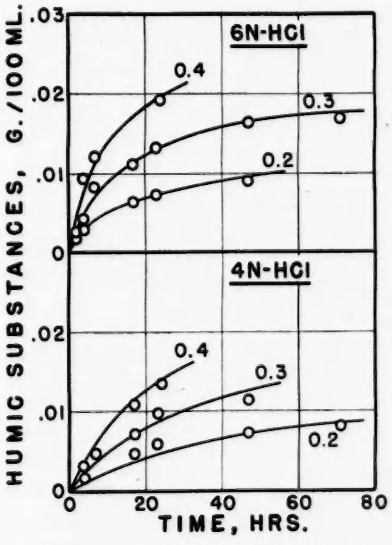

FIG. 2. *The rate of formation of humic substances from glucose at various concentrations, in 6N and 4N hydrochloric acid, at 100°C. The circles are the experimental points; the curves drawn through them were obtained with the aid of equation (5) as described in the text. The figures near the curves refer to the initial glucose concentrations in g./100 ml.*

Since decomposition of glucose is a monomolecular reaction [13, 14, 20] whereas the formation of humic substances is of a different order, it is obvious that the over-all reaction must occur in at least two steps with at least one intermediate product (which might well be the pre-humin designated by Schweizer [22] as hymatomelanic acid, a dehydration product of glucose). If the formation of humic substances from pre-humin is a condensation reaction, it may obey approximately the equation of a bimolecular reaction:

$$kt = \frac{h}{p(p - h)},$$

or

$$h = \frac{p^2}{\frac{1}{kt} + p}, \tag{3}$$

where p = initial concentration of pre-humin, h = concentration of humic substances at time t, and k = the rate constant. If it is assumed that pre-humin is formed in a relatively fast reaction from only a fraction of the glucose present (the remainder furnishing soluble decomposition products such as ω-oxymethylfurfural, levulinic and formic acids), the initial concentration of pre-humin, p, may be expressed as

$$p = fg, \tag{4}$$

where f = a proportionality constant, representing the fraction of glucose transformed into pre-humin, and g = initial glucose concentration. Combining equations (3) and (4),

$$h = \frac{f^2g^2}{\frac{1}{kt} + fg}. \tag{5}$$

Using experimental values of h at various times t for constant initial glucose concentration g in equation (5), the constants f and k were evaluated. The constant f was found to be independent of acid concentration, glucose concentration, and temperature, and to have the value 0.07. If the concentrations in equations (3), (4), and (5) are expressed in g./100 ml., the rate constants k obtained for different acid concentrations and temperatures are those listed in the third column of Table III. Using these constants in equation (5) to compute h, smooth curves for 6*N* and 4*N* hydrochloric acid at 100°C were plotted in Figure 2. Agreement between these cal-

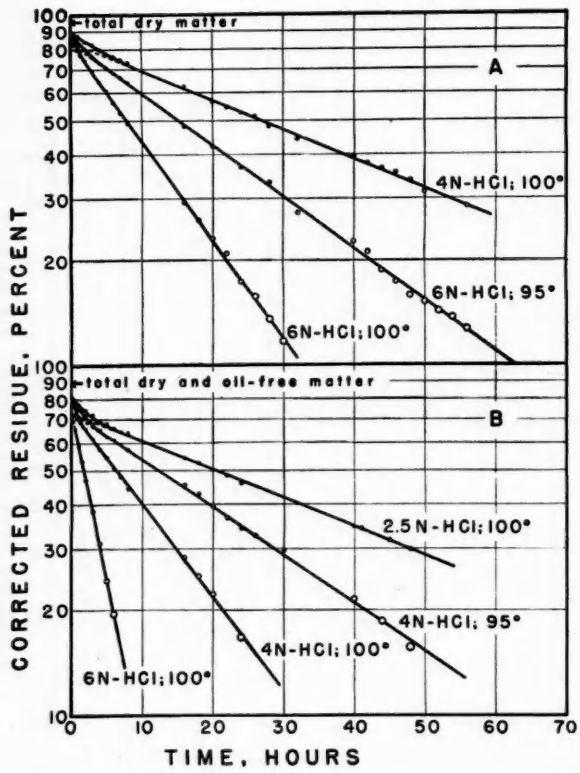

FIG. 3. *The effect of acid concentration and temperature on the hydrolysis of cotton linters* (A) *and Fortisan rayon* (B).

culated curves and experimental points is quite satisfactory, especially since equation (5) is to be used only to compute small corrections for humic substances in the observed cellulose hydrolysis residues.

Since the rate of humic-substance formation from glucose was known, the amount formed during the hydrolysis of cellulose could be determined. The formation of humic substances may be regarded as a two-step reaction, the first step leading from cellulose to glucose and the second from a portion of the glucose to the humic substances. Consecutive reactions, with one of the first order followed by one of the second order, lead to a complicated integral. In addition, the rate constant of the first step in this case is not known. Therefore, the humic-substance content of the residues was determined in the following stepwise manner.

In first approximation, the total observed residue (cellulose plus humic substances) at any time t is considered to be unhydrolyzed cellulose. Then the expression $G = 1.11\ (C-r)$ gives the total amount of glucose, G, produced up to that time, when C is the total cellulose in the sample (determined analytically), r is the observed residue at time t, and the factor 1.11 is required because, theoretically, upon complete hydrolysis 1 part of cellulose yields 1.11 parts of glucose. Substituting the above value of G for g in equation (4), the total amount of pre-humin, P, formed up to time t is

$$P = 1.11f(C-r) = 0.078(C-r). \qquad (6)$$

Since additional amounts of pre-humin are formed from cellulose during each time interval, and part of the total amount produced has been transformed into humic substances previous to that period (except for the first period), the initial concentration of pre-humin, p, for any particular time interval is set equal to

$$p = \bar{P} - H, \qquad (7)$$

where $\bar{P}$ is the arithmetic mean of the total amounts of pre-humin produced at the beginning and at the end of the particular time interval (that is, $\bar{P} = (P_1 + P_2)/2$, the subscripts referring to the beginning and the end of the time interval, respectively; and H is the total humic correction up to the beginning of that time interval. Substitution of the above value of p in equation (3) gives h, the amount of humic substances formed during that interval. Successive application of equations (6), (7), and (3) to all of the time intervals employed, and the successive accumulation of h, yields a series of corrections for humic substances, H. These are subtracted from the observed residues, r, to yield the corresponding amounts of unhydrolyzed cellulose.

It is preferable to express these corrections on the same basis as the hydrolysis-residue values—that is, in percentage of original sample weight. Expressing the concentrations in equations (3), (6), and (7) on that basis, and keeping cellulose sample weight and acid volume constant at 0.2 g. and 50 ml., respectively, the constant k assumes the values given in the last column of Table III for various acid concentrations and temperatures.

Upon subtraction of the corrections for humic substances from the observed residues and recalculation with the corrected residues, it is found that the second approximation does not change the first decimal of the former. The first approximation is, therefore, com-

TABLE IV. RESULTS OF HETEROGENEOUS HYDROLYSIS OF COTTON LINTERS AT VARIOUS ACID CONCENTRATIONS AND TEMPERATURES

Temperature	100°C		95°C
HCl concentration	4*N*	6*N*	6*N*
Crystalline cellulose, c_o (%)	84.0±0.6	83.3±0.5	83.8±0.6
Amorphous cellulose, a_o (%)	7.0±0.5	7.7±0.5	6.4±0.5
Total cellulose, C (%)	91.0±0.8	91.0±0.7	90.2±0.8
Rate constant, k_c (hours^{-1})	0.0192	0.0646	0.0338
Rate constant, k_a (hours^{-1})	0.365	1.126	0.670

pletely satisfactory. Since most of the time intervals are of 1-hour duration, the calculation of this correction can be greatly simplified if a table of h as a function of p according to equation (3) is prepared for $t = 1$ and for various rate constants k.

Application of the corrections for humic substances to the hydrolysis-residues of cotton D and plotting of the corrected residues on a semilogarithmic scale against time gave the continuous curve of Figure 1. This curve, typical of all the other cellulose samples examined, has the character demanded by equations (1) and (2) for cellulose hydrolysis curves—that is, after an initial curvature, corresponding to a more rapid hydrolysis, the curve passes over into a perfectly straight line corresponding to the slower monomolecular hydrolysis of the crystalline cellulose. Although the influence of the correction for humic substances becomes significant only at an advanced stage of hydrolysis, application of this correction is necessary to permit an unambiguous linear extrapolation to zero time for the determination of crystalline cellulose.

The validity of this correction is confirmed by further work, yet to be published.

Effect of Acid Concentration and Temperature

To study the effect of acid concentration and temperature, samples of cotton linters and of Fortisan rayon were hydrolyzed with hydrochloric acid of different concentrations at 100° and at 95°C for various lengths of time. The results are shown in Figure 3, in which the corrected weights for unhydrolyzed cellulose, expressed in percents of the original, are plotted against time on a semilogarithmic scale. It may again be seen that after an initial rapid reaction the curves become linear as demanded by theory (equations (1) and (2)). Linearity is maintained even when less than 10 percent of the original sample remains unhydrolyzed. The rate of hydrolysis increases with increasing acid concentration and temperature, but the extrapolated straight lines pass within the limits of error through the same point on the vertical axis.

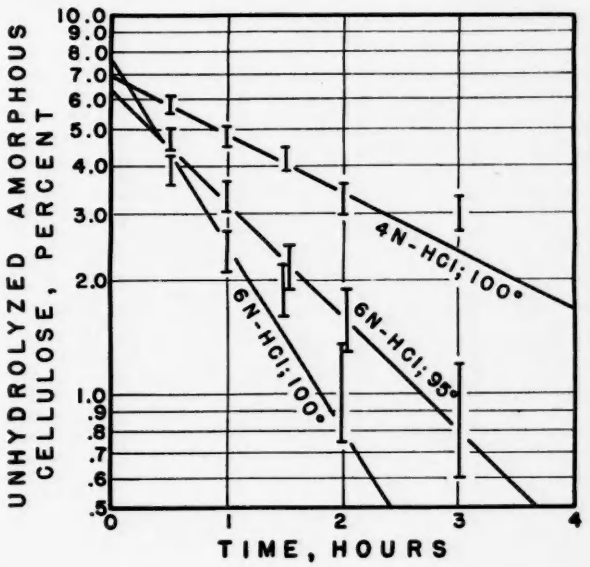

FIG. 4. *The hydrolysis of the amorphous cellulose in cotton linters at various acid concentrations and temperatures.*

When the differences between the curved and the extrapolated linear parts of the hydrolysis curves of Figure 3A are plotted as a function of time on a semilogarithmic scale, the graph of Figure 4 is obtained, which shows the hydrolysis of the amorphous part of the cellulose in cotton linters. Since the points in Figure 4 represent small differences between large figures, they contain appreciable errors but, nevertheless, they lie on straight lines in accordance with theory.

Although graphical extrapolation would be entirely satisfactory, the equations for the linear parts of the hydrolysis curves were calculated by the method of least squares and extrapolated mathematically in order to obtain an estimate of the precision of the method. The results are presented in Table IV for cotton linters and in Table V for

TABLE V. RESULTS OF HETEROGENEOUS HYDROLYSIS OF FORTISAN RAYON AT VARIOUS ACID CONCENTRATIONS AND TEMPERATURES

Temperature	100°C	100°C	100°C	95°C
HCl concentration	2.5N	4N	6N	4N
Crystalline cellulose, c_o (%)	73.1±0.5	73.6±0.8	73.6±0.5	73.8±0.4
Amorphous cellulose, a_o (%)	8.8±0.4	8.0±0.9	—	8.0±0.8
Total cellulose, C (%)	81.9±0.6	81.6±1.1	—	81.8±0.9
Rate constant, k_c (hrs.$^{-1}$)	0.0186	0.0607	0.2203	0.0313
Rate constant, k_a (hrs.$^{-1}$)	0.396	1.257	—	0.641

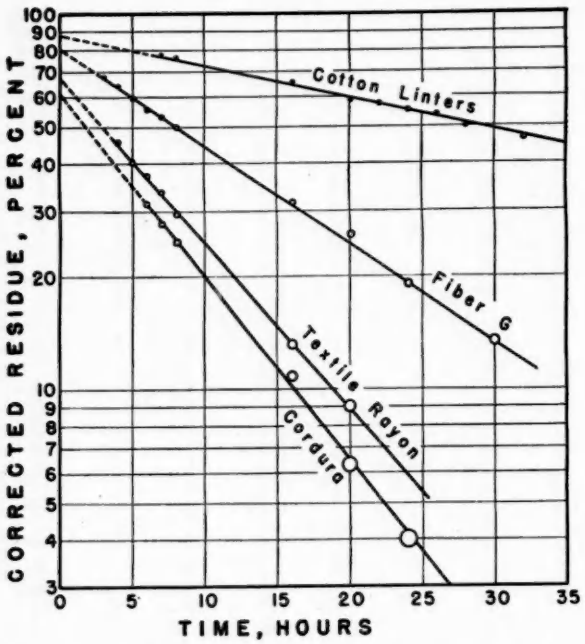

FIG. 5. *Hydrolysis curves of several cellulosic materials with 4N hydrochloric acid at 100°C yielding different degrees of crystallinity.*

Fortisan rayon. With 6*N* acid the hydrolysis of Fortisan proceeded so quickly that not enough points could be obtained for the estimation of amorphous cellulose. It is apparent from the data that neither acid concentration nor temperature has any effect on the amount of crystalline or amorphous cellulose as determined by acid hydrolysis.

The hydrolysis of the amorphous cellulose proceeds roughly about twenty-one times faster than that of the crystalline cellulose, as shown by the rate constants in Tables IV and V. From the rate constants k_c of the crystalline cellulose at 95° and 100°C, the energy of activation, E, of the hydrolysis of crystalline cellulose, computed by the Arrhenius equation,

$$\ln k_c = \frac{-E}{RT} + \text{constant},$$

is:

Cotton linters ..... $E = 34.6$ kcal.
Fortisan rayon ..... $E = 35.4$ kcal.
Average ..... $E = 35.0$ kcal.

These values are lower than those obtained by Saeman [20], who found 42.9 kcal. between 170° and 190°C, and higher than those of Eisenhut and Schwartz [7], who found 27.8 kcal. for the temperature interval of 35° to 55°C. The discrepancies are probably due to the different temperature at which the activation energies were determined. All of the figures quoted above apply to the heterogeneous hydrolysis of cellulose. For the hydrolysis in the homogeneous phase between 12° and 40°C, activation energies of 29.8 kcal. [8] and 29.6 kcal. [21] are reported.

The rate constants k_a of the amorphous cellulose are based on only few and relatively inaccurate data (see Figure 4) which are not reliable enough for computation of the activation energy.

Determination of Relative Proportions of Crystalline and Amorphous Cellulose

If allowance is made for moisture in purified linters and for moisture and oil in Fortisan, one arrives at a total cellulose content of 95.5 percent for the former and 89.0 percent for the latter. These values are higher than the ones obtained by the hydrolysis method (see Tables IV and V). The deficiency appears to be due to solution of the shorter cellulose molecules of the original sample in the hydrolyzing agent. As the hydrolysis curves in Figure 3 show a great loss of weight during the first half-hour, the solution of short chains must take place at a very early stage of the reaction and hence almost solely at the expense of the more accessible amorphous cellulose.

Because of this loss of amorphous cellulose at an early stage of hydrolysis and because of the relatively great uncertainty in determining the amorphous cellulose content by extrapolation of data obtained by subtraction of large figures, it appears preferable to determine only the crystalline cellulose content by the hydrolysis method. The amorphous cellulose content may then be obtained by subtracting the amount of crystalline from the total amount of cellulose as determined by an independent method.

The hydrolysis method of estimating the relative proportions of crystalline and amorphous cellulose described above was applied to a variety of cellulosic materials, as listed in Table VI. Since it had been established that neither temperature nor acid concentration influenced the crystalline-amorphous ratios, the hydrolysis of all the cottons was carried out with 6*N* HCl and that of the faster-hydrolyzing rayons with 4*N* HCl, both at 100°C. Hydrolysis-residues were usually determined at 1-hour intervals

TABLE VI. RESULTS OF HETEROGENEOUS HYDROLYSIS OF VARIOUS CELLULOSIC MATERIALS

Material	Crystalline cellulose on dry basis (%)	Total cellulose on dry basis (%)	Degree of crystallinity	Rate constant, k_c, at 100°C 4*N* HCl Hrs.$^{-1}$	6*N* HCl Hrs.$^{-1}$
Ramie	77.8±0.8	81.6	0.95	—	0.0731
Raw cotton A	80.1±0.7	94.5	.85	—	.0782
Raw cotton B	78.0±0.6	93.2	.84	—	.0794
Raw cotton C	78.7±0.5	95.2	.83	—	.0835
Raw cotton D	83.2±0.3	95.1	.88	—	.0787
Raw cotton E	82.7±0.4	98.1	.84	—	.0754
Raw cotton F	81.5±0.5	96.7	.84	—	.0743
Raw cotton G	78.7±0.7	96.5	.82	—	.0787
Raw cotton H	82.4±0.4	96.8	.85	—	.0794
Raw cotton I	83.1±0.5	95.7	.87	—	.0826
Purified cotton linters	87.6±0.6	100.0	.88	0.0192	.0646
Mercerized cotton B1	76.5±0.6	97.6	.78	—	.0879
Mercerized cotton B2	66.9±0.4	98.2	.68	—	.0731
Fortisan rayon	82.6±0.6*	100.0*	.83	.0607	—
Fiber G rayon	81.2±0.5	100.0	.81	.0596	—
Cordura rayon	61.8±0.8	100.0	.62	.1120	—
Textile rayon	67.9±0.5	100.0	.68	.1020	—
Special rayon	69.1±0.7	100.0	.69	.0833	—

* This value is based on the total dry and oil-free matter and not on the dry matter only.

for 8 hours and at 16, 20, 24, and 30 hours. After application of the correction for humic substances the corrected residues were plotted against time on semilogarithmic graph paper. The linear part of these plots was extrapolated to zero time by the method of least squares, which gave the percentage of crystalline cellulose in the air-dry sample and, after adjustment for moisture content, the percentage of crystalline cellulose on a dry basis. The data thus obtained, together with their standard errors, are presented in the second column of Table VI. The linear parts of the hydrolysis curves of some of the materials of Table VI are shown in Figure 5.

For the determination of the percentage of amorphous cellulose and for a comparison between materials such as cotton and ramie, which also contain noncellulosic constituents, it is desirable to express crystallinity on the basis of total cellulose rather than of total dry matter. For this reason the "degree of crystallinity" was defined as the amount of crystalline cellulose in a sample expressed as a decimal fraction of the total cellulose content. The degree of crystallinity, together with the total cellulose content on which it is based, is also given in Table VI. For all the rayons and for purified linters, total cellulose was set equal to total dry weight, except in the case of Fortisan, for which the total of dry and oil-free matter was used. For cotton and for partly purified ramie, cellulose contents were determined analytically by the method of Kettering and Conrad [12], the sole variation being that the isolated cellulose was weighed instead of titrated. In the last column of Table VI are reported the rate constants determined for the hydrolysis of crystalline cellulose.

Discussion of Results

The data reported here indicate that there are two distinct rates of hydrolysis, which have been attributed to the rapid hydrolysis of the amorphous portion and the slower hydrolysis of the crystalline portion of the fiber. Assuming this interpretation to be correct, the hydrolysis method offers a procedure for determining the relative proportions of crystalline and amorphous cellulose which has several advantages: it is experimentally simple; it is capable of straightforward interpretation of the observed data; and, although like all chemical methods it requires an extrapolation, the extrapolation is unambiguous because the plotted function is linear, even when the reaction is carried almost to completion.

In principle, this method of interpretation is also applicable to the hydrolysis and catalytic oxidation procedure [5, 16–19], which measures the amount of cellulose hydrolyzed as a function of time. However, this procedure depends on a constant rate of evolution of carbon dioxide from glucose, which actually prevails only during the early stages of the

reaction. It is, therefore, not possible to follow the course of hydrolysis to, and hence not beyond, the point at which the hydrolysis plots become linear. For example, the rate of hydrolysis of cotton linters could previously [19] be measured only to a point where 24 percent of the original sample was hydrolyzed, whereas the present method showed (see Figure 3A) that all amorphous cellulose had not disappeared until 25 to 30 percent of the original linters had been hydrolyzed.

The summary of results (Table VI) obtained by application of the new hydrolysis method shows that ramie, the cellulose content of which is 95 percent crystalline, has the highest degree of lateral order of all cellulose fibers on which determinations were made.

The raw cottons examined range in degree of crystallinity from 0.82 to 0.88. While samples A, B, and C represent three different varieties, cottons D to I are of the same variety but vary in their degree of orientation (see Table I). It may be seen from Table VI, in which the cottons D to I are arranged in order of decreasing orientation, that there is no correlation between the degree of orientation and the degree of crystallinity, and that degree of crystallinity varies more among different cotton samples of the same variety than among the different varieties studied.

Purified cotton linters were found to contain as much crystalline cellulose as, or more than, some of the raw cottons. This is not surprising in view of their rather severe purification treatment, which may remove some of the less-resistant amorphous cellulose together with noncellulosic constituents.

The effect of mercerization on crystallinity is illustrated by the two samples of mercerized cotton B1 and B2. Under the influence of the swelling action of the strong alkali and in the absence of any tension, the degree of crystallinity of raw cotton B is reduced from 0.84 to 0.68. This corresponds to a doubling of the amount of amorphous cellulose and places the "shrunk-mercerized" cotton in the same crystallinity group with some of the rayons. If, however, mercerization is carried out under maximum tension, as in the preparation of sample B1, then some of the disordering influence of the strong alkali is counteracted by the tension, and crystallinity decreases only from 0.84 to 0.78.

The five rayons listed in Table VI show great differences in degree of crystallinity. This property

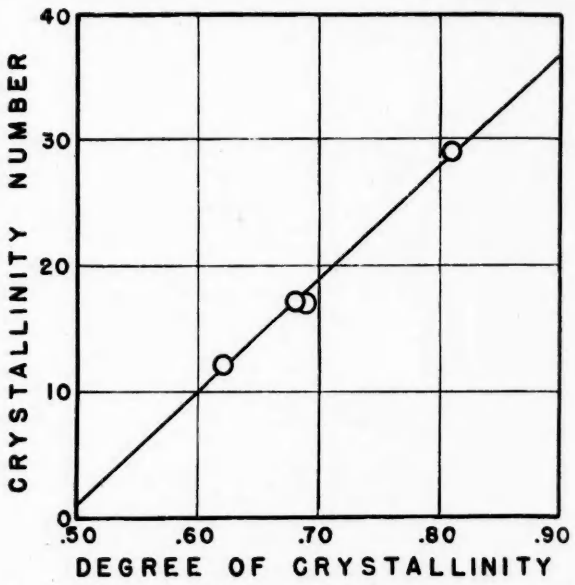

FIG. 6. *Relationship between the degree of crystallinity determined by hydrolysis and the crystallinity number derived from x-ray patterns.*

is compared with other physical properties in Table VII. Tenacity, ultimate elongation, and moisture content were determined at 70°F and 65 percent relative humidity after preconditioning at a relative humidity of 50 percent. The "crystallinity number" of four of the rayons is derived from x-ray diffraction patterns and is defined [5] as the difference between the average intensity of the 101 interference and that of the minimum between the 101 and $10\bar{1}$ interferences, expressed as a percentage of the average intensity of the 101 interference. It is a measure of crystallinity on an arbitrary scale.

Fortisan and Fiber G, in whose manufacture extreme stretching is employed, show the highest degree of crystallinity. Ordinary textile rayon, which is only moderately stretched, and the special rayon, prepared without any appreciable tension, have a much lower degree of crystallinity. Despite its greater strength, textile rayon contains about the same percentage of amorphous cellulose as does the special rayon. Finally, Cordura, a high-tenacity rayon made especially for use in tire cord, contains the largest amount of amorphous cellulose of any of the five rayons. Obviously there is no correlation between the degree of crystallinity and the tenacity of rayon. The latter seems to be controlled primarily by the degree of orientation of the crystallites.

Apparently, during the formation of the rayon filament and the subsequent drying an appreciable

quantity of crystallites is formed even without stretching, as may be seen from the degree of crystallinity of the special rayon. However, the degree of crystallinity seems to depend also to a large extent on the particular conditions maintained during spinning, since high-tenacity Cordura contains less crystalline cellulose than low-tenacity textile rayon, and the moderately stretched textile rayon contains about the same amount as the unstretched special rayon.

With one exception, there is a tendency for the ultimate elongation to increase with increasing percentage of amorphous cellulose, which is quite plausible since greater amounts of amorphous cellulose in the fiber should permit greater elongation. The extremely high elongation of the textile rayon may be due to other factors. There is also a very slight tendency for the moisture content to increase with an increase in the relative proportion of amorphous cellulose, because amorphous cellulose has a greater affinity for moisture than crystalline. However, this effect is small, possibly because of the high relative humidity at which it was determined.

The degree of crystallinity determined by the hydrolysis method and the crystallinity number derived from x-ray diagrams are definitely correlated, as shown by Figure 6, where the crystallinity number is plotted as a function of the degree of crystallinity for the four rayon samples for which both are available. This furnishes additional evidence of the validity of the results obtained by the hydrolysis method. Although many more data will have to be collected before any real correlations between the degree of crystallinity and other physical properties can be established with certainty, the data in Table VII may serve to indicate possibilities. For example, it might be expected that the degree of crystallinity may have an important influence on the flex-fatigue life of synthetic and natural fibers.

TABLE VII. COMPARISON OF SEVERAL PHYSICAL PROPERTIES OF FIVE SAMPLES OF RAYON

Material	Degree of crystallinity	Tenacity (g./den.)	Ultimate elongation (%)	Moisture (%)	"Crystallinity number"*
Fortisan	0.83	7.6	5.9	8.2	—
Fiber G	0.81	5.0	7.2	10.2	29
Special	0.69	0.3	8.2	11.6	17
Textile	0.68	2.5	15.8	11.3	17
Cordura	0.62	3.9	9.4	11.6	12

* This figure is derived from x-ray diagrams and is defined as the difference between the average intensity of the 101 interference and that of the minimum between the 101 and $10\bar{1}$ interference, expressed as a percentage of the average intensity of the 101 interference.

The degree of crystallinity found for linters and ramie by the new hydrolysis method is in agreement with data obtained by the thallous ethylate method for swollen and dried samples, but is very much lower than that reported for unswollen, dried samples, for both of which a crystalline cellulose content of over 99 percent is reported [2]. However, the conclusion that the hydrolyzing agent leads to appreciable swelling of the cellulose does not seem justified because there is little probability that amorphous cellulose is almost absent from unswollen linters and ramie in view of the diffuse background found in x-ray diagrams of such materials. Furthermore, treatment of cellulose with hydrochloric acid in a heterogeneous system produces no changes in the spacings of the x-ray diffraction pattern of cellulose; hence the acid seems not to swell the crystalline portions. No practicable comparison with results obtained by the hydrolysis and catalytic oxidation method is possible because, as has been pointed out, a strictly quantitative evaluation of these data cannot be made. For the oxidation of cellulose with periodic acid, amorphous cellulose contents of 6.0 percent are reported [9] for purified cotton linters, and 1.1 to 2.1 percent for cotton. It is possible that incompleteness of reaction and uncertainty of extrapolation, based on only three points, are responsible for these low figures.

The last three columns of Table VI reveal that there is no connection between the degree of crystallinity and the rate of hydrolysis of the crystalline cellulose. It is noteworthy that reaction rates are similar for the cellulose from naturally grown fibers, regardless of its origin, or whether it is native or converted to hydrate cellulose. Thus, the rate of hydrolysis of the crystalline portion of mercerized cotton is about the same as that of the same cotton before mercerization, despite the appreciable swelling during mercerization and the great change in crystallinity resulting from this treatment. On the other hand, the regenerated fibers have a much greater rate of hydrolysis and their rate constants show substantial differences. No satisfactory explanation of this observation can be offered at this time.

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Mildew- and Rot-Resistance of Textiles

P. B. Marsh*

MUCH interest was manifested during World War II in the matter of prevention of mildewing and rotting of fabrics and other textile items for use by the Armed Forces of the United States and her Allies. Rotting was a problem with sandbags used for barricade purposes. Tents were exposed in moist tropical climates conducive to microbiological fiber deterioration. Military fabric items of many kinds were often of necessity treated very roughly in use, being commonly smeared with mud, rolled up and packed wet, or left for long periods in poorly ventilated storage. U. S. Army procurements included more than 250,000,000 yards of rotproofed sandbag fabric [57], millions of yards of mildewproofed tent fabric [59, 82], and large amounts of mildewproofed material for jungle hammocks [59], insect netting [59], and camouflage nets. Major amounts of mildewproofed webbing, thread, and rope were also used.

Although a number of important problems relating to the mildew-resistance of textiles and textile fibers are still only partially solved, definite progress has been made in this field both during and since the war and certain problems for the future have been clearly outlined. The purpose of this article is to summarize the most important published information relating to mildew- and rot-resistance of textiles and textile fibers. It is intended both for the reader interested only in an evaluation of the present status of knowledge of the subject and for the experimentalist interested in further research in the field.

Causes of Textile Deterioration

In out-of-door exposures, fabrics and other textiles made of either natural or synthetic fibers commonly undergo tendering or decrease in strength. In the case of articles made of cotton or other plant fibers or of the regenerated-cellulose rayons, this tendering is frequently very rapid if the articles rest in contact with moist soil or if they become extensively contaminated with soil and are then exposed to dampness. Under these circumstances the fibers are decomposed or rotted by soil microorganisms. With military textiles soil-contact exposures are very common, particularly in combat areas and during troop movements. A fabric carrying case containing a "walkie-talkie" may have to be placed in the bottom of a muddy foxhole. A tent may be dragged across wet and muddy ground. Portable military water reservoirs are often set up in wet situations in direct contact with soil. The sandbag faces soil-contact exposures in both war and peace. While perhaps not designed for such use, tarpaulins are also often seen even in peacetime service in contact with soil.

Above-ground weather exposures without soil contact or extensive soil contamination may result, under certain conditions, in severe microbiological deterioration, although in general this tendering is less rapid than that which occurs in soil contact. Such deterioration may occur, for example, in the case of tents, tarpaulins, or awnings. Rapid fabric tendering of nonbiological origin is also of frequent occurrence in above-ground exposures, especially in the presence of moderate-to-intense sunlight. Nonbiological textile deterioration in sun exposures is apparently largely a result of photochemical degradation, and is localized especially at the exposed surfaces [29]. Fargher [42] states that thin, open-weave fabrics in which a high percentage of the individual fibers are exposed to light are more susceptible to nonbiological damage in above-ground exposure than are thicker close-weave fabrics; with fabrics of the latter type, in which the surface fibers shield and protect underlying fibers as well as fiber-deteriorating fungi from the destructive effects of light, tendering is believed to be in general more predominantly a result of biological decay. Barnard [12] records a suggestion by Dean to the effect that with relatively thick or closely woven fabrics the slower rate of drying after periods of wet weather is probably conducive to biological deterioration.

Microbiological Agents

The ability to decompose cellulose is very common among the thousands of species of nonparasitic or mold fungi. Cellulosic fibers, either natural or

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regenerated synthetic, are thus vulnerable to attack by many different fungi. The susceptibility of wool and silk to decay and tendering by microorganisms has not been very thoroughly investigated, although in general they appear to be susceptible to fewer organisms than the cellulosic fibers are. Such information as is available concerning the mildew-resistance of these noncellulosic fibers is discussed later in this paper (page 603).

Present-day knowledge of the occurrence of cellulose-decomposing species among the fungi has been contributed over a period of years by numerous authors [61, 62, 78, 92, 97, 105, 115, 134, 138], the most important single investigation on the subject being one by White and his coworkers [138]. All of the available evidence indicates that in certain common genera, such as Chaetomium, Trichoderma, Memnoniella, Stachybotrys, and Alternaria, most, if not all isolates are active cellulose-decomposers, whereas in other genera, such as Aspergillus and Penicillium, activity is limited to certain species. In still other genera activity is found only very infrequently or not at all. The genera of the Mucorales—for example, Mucor, Rhizopus, Absidia, Circinella, etc.—have been found to be notably lacking in cellulose-decomposers [97, 128, 138]. Quantitative data on rates of tendering of cotton fabric by different fungi under pure-culture laboratory conditions are contained in a number of papers [61, 62, 78, 130]. According to the results described in these papers, many fungi are capable of deteriorating a lightweight canvas so rapidly under ideal growth conditions in the laboratory that it will have no measurable strength left after a 2-week exposure; a few of them can produce this result within one week or even less.

Much interest attaches to the question of which fungi are most important in textile deterioration. Microscopic observations of the deteriorated textile articles concerned are of course necessary in seeking the answer to this question. Broughton-Alcock [24] reported Stemphylium to be the primary cause of damage to deteriorated tents on the Island of Malta during World War I. Neill and Travers [90] found Stemphylium and Alternaria (these are closely related forms) on almost all samples of cotton duck exposed above-ground under jungle conditions; *Cladosporium herbarum* was exceedingly common and Pleospora (possibly a form in the life cycle of Alternaria) and Phoma species were found in advanced stages of fabric deterioration. Zuck and Diehl [142], in general agreement with Neill and Travers [90], reported an early growth of Alternaria on cotton duck in above-ground exposure, followed after a period of several months by Phoma (including its close allies Diplodia, Hendersonia, and Diplodiella) and Leptosphaeria, forms apparently adapted to surviving the adverse effects of light and desiccation commonly encountered by fabrics in above-ground exposure. The importance of these same weather factors in determining the fungus flora of dew-retted flax and hemp has been pointed out by Jensen [77] and by Fuller and Norman [48]. Bayley and Weatherburn [17], in accord with other observations just mentioned, found Alternaria as an early growth on cotton fabric exposed on weathering racks. In soil-contact exposures a wide variety of different fungus forms have been observed to grow on cotton fabric, including a number of hyaline or light-colored forms not as yet shown to occur on fabric in moderate-to-high-light, above-ground exposures—for example, Trichoderma, Fusarium, Penicillium, Aspergillus. Observations of fungi growing on cotton fabrics, particularly under storage conditions, mention the fungus *Memnoniella echinata* with such frequency [1, 83, 137] that one is tempted to postulate the existence of some unusual adaptive physiological mechanism in this genus. The ability of the fungus to grow luxuriantly on cotton duck in high-humidity exposure without the addition of liquid water has been shown by experiments in the author's laboratory [84] and furnishes a clue suggesting a possible cause for its widespread occurrence. On deteriorated cordage in marine exposures in various Massachusetts harbors, Barghoorn and Linder [10] found *Halophiobolus salinus* to be of frequent occurrence; this organism, as well as several others described in the same article, appears to be especially adapted to growth under salt-water conditions.

In most recent work relating to the growth of fungi on fabrics primary emphasis has been placed on actual tendering. Reports from military sources suggest, however, that the activity of nontendering organisms may also be important in some instances. It is commonly stated, for example, that certain fungi may grow on water-repellent wax treatments on fabrics, thereby lowering the fabrics' resistance to wetting and penetration by water. In other instances it is believed that copper-tolerant nontendering forms actually grow on mildew-preventive copper soaps of

the nature of copper stearate or copper oleate, thereby rendering them water-soluble and leaving the fiber unprotected against the subsequent action of less copper-tolerant cellulose decomposers. This latter phenomenon probably occurs especially in soil-contact exposures.

The relative importance of the bacteria as compared with the fungi in the deterioration of textiles and textile fibers is not completely understood, although one may reason from the known high moisture requirements of the bacteria that they might be expected to be of primary importance only under aquatic or very moist situations. Fish-net deterioration, for example, is stated by some writers to be bacterial in nature [107] although even here there is evidence to indicate that fungi may be of great importance [10]. With the exception of the cytophagas, the cellulose-decomposing bacteria have not been readily isolated or maintained in culture and have been found generally to be more particular in their growth requirements than the cellulose-decomposing fungi; some of them require anaerobic conditions [96], others high temperatures, and many a relatively high hydrogen ion concentration [40].

Among the aerobic cellulose-decomposing bacteria, *Sporocytophaga myxococcoides* (known earlier as *Spirochaeta cytophaga*) has been the most thoroughly investigated. It has been shown by several authors [78, 104, 130] to decompose cellulose very rapidly under suitable conditions. The erratic nature of its growth, as discussed by Thom *et al.* [130], appears, according to the observations of Thaysen and Bunker [128, p. 46] and the later carefully conducted experiments of Reese [104], to be related to its extreme sensitivity to relatively slight differences in the pH of the growth medium and possibly also to its requirements for iron. "Organism B" of Reese [104] was likewise exacting in its growth requirements. A number of aerobic mesophilic bacteria have been claimed to be cellulose decomposers on the basis of what appears to be inadequate or at least questionable evidence, particularly if one uses the term "cellulose" in the more restricted sense. The subject is discussed by Thaysen and Bunker [128, pp. 47–53]. Fuller and Norman [47] have shown that xylan is much more readily decomposed by aerobic mesophilic bacteria than is cellulose. The ability to bring about cellulose decomposition in terms of digestion of Norman's "natural celluloses" [93, p. 29] in plant residues is, as that author points out, by no means the same thing as the ability to decompose cellulose in the form of cotton or filter paper.

There is evidence to suggest that, at least under some conditions, wool is susceptible to attack by certain bacteria. The subject is considered on page 603.

The cellulose-decomposing enzyme or enzyme complex known as "cellulase" has never been isolated in pure condition and the few papers pertaining to it are fragmentary and generally unsatisfactory. To the author's knowledge no one has yet published evidence demonstrating more than very slow cellulase activity in extracts proved to be free of living cells. Evidence, perhaps more suggestive than conclusive in nature, has been offered by Pringsheim [101] and later by Simola [114] to show that one of the intermediates in the enzymatic breakdown of cellulose is the disaccharide cellobiose.

Nonbiological Agents

Nonbiological deterioration of sun-exposed cotton fibers or fabrics has been reported by Holman and Jarrell [67], Jarrell, Stuart, and Holman [75], Grimes [63, 64], Fargher [42], Barghoorn [9], Race and Rowe [102], Dean and Worner [36], and others. Conn made similar observations on linen [30]. Of the two types of deterioration—microbiological and nonbiological—either may be severe, depending on the exposure conditions. Light-shielding pigments, such as were used during the recent war in the U. S. Army's fire-, water-, and weather-resistant tent duck [73, 80], appear to decrease the extent of light damage to fabric [9, 74]. The use of inorganic pigments in fiber coating compositions to retard light-deterioration of fibers and to counterbalance the stimulative effects of certain coating compounds on photochemical fiber deterioration has been described by Holman and Jarrell [68], Jarrell and Holman [74], and Leatherman [80]. It is generally agreed that chromium oxide deposited on the fiber slows down nonbiological fiber deterioration in light [19, 42, 75, 102]. Evidence from experiments described by Barghoorn [9] indicates that various mildew-preventive agents may materially increase the susceptibility of fabric to light-deterioration. The conditions under which this accelerated tendering in the presence of light occurs are not adequately understood. Bayley and Weatherburn [19] found that a petrolatum-containing waterproofing wax stimulated nonbiological breakdown of fabric to a marked degree.

Jarrell, Stuart, and Holman [75] noted that for some unexplained reason "the organism *Chaetomium globosum* does not attack cotton fibers which have become degraded and weakened by sunlight to the same extent as it attacks new, unweathered cotton fibers." The data of Wagner, Webber, and Siu [133] suggests the existence of a similar phenomenon with cotton exposed in the laboratory to ultraviolet radiation and subsequently to *Metarrhizium glutinosum* (= *Myrothecium verrucaria*). Neither group of authors suggests, of course, that any retarding effect of ultraviolet exposure on subsequent microbiological deterioration would overbalance the primary tendering effect of light.

Effects of Microbiological Deterioration on Textile Properties

In above-ground out-of-door exposures one of the first symptoms of microbiological deterioration of a textile fiber is a spotting or general darkening in color. Even before it is picked, cotton in the boll frequently develops a so-called "blue stain" in moist weather which is due to the growth of fungi [95]. In the case of sun-exposed cotton fabrics the darkening or spotting takes place primarily on the under surface of the fabric [36, 142]. The spots are sites of fungus colonies and their dark color has been suggested to be due, at least in part, to lignin or lignin-like complexes in the walls and spores of the fungi involved [141]. One observation is on record of a shade exposure in which a light-colored, lignin-decomposing fungus actually overgrew an earlier dark-colored invader and brought about a bleaching of the color [141].

Under humid storage conditions textiles frequently develop a musty odor, which is a reliable criterion of microbiological action [100]. Mildew spots on fabrics in storage may assume a diamond shape owing to the ease with which the fungi grow in the directions of the warp and woof of the fabric [54]. Fargher [42] finds fungus growth on "gray" cotton in high-humidity exposure to be accompanied by a rise in pH. In some cases pH increases of 2.5–3.0 pH units were observed. Galloway and Burgess [56, p. 132] record a similar observation, although without citing detailed evidence.

Under soil-contact conditions one of the early symptoms of fungus deterioration is the clinging of soil particles to the fiber when it is removed from the soil, this being due to the binding of such soil particles to the fiber by the hyphae or threads of the fungus itself.

Pronounced strength losses of microbiological origin have been noted with fibers and textiles under a variety of different situations. Contact with warm, moist soil is generally by far the most favorable of these situations for rapid fiber-strength losses of microbiological origin. Over-retting of flax or hemp, in which strength losses can be caused by pectin-dissolving as well as cellulose-decomposing organisms, is also in some cases very rapid [77, 128, p. 170]. Zuck and Diehl [142] have measured microbiological strength losses in sun-exposed cotton duck. Brittleness is particularly noted by Denham [37] as a property of biologically deteriorated cotton fibers; it is likewise noted by Serrano [112] in the case of biologically deteriorated abaca or "Manila hemp" and by Finlow [44] with "heart damaged" jute.

Fibers lose weight under the influence of microbiological deterioration. Exactly how much weight they lose, however, is not easily determined with accuracy because part of the fiber material decomposed is converted into fungus substance and no convenient way appears to have been devised to determine quantitatively the amount of fungus material in a deteriorated fiber. Such data as are presented on this point by Rogers, Wheeler, and Humfeld [108], combined with the relatively sparse appearance of fungus mycelium in certain highly tendered fibers, lead one to believe that at least under some circumstances a relatively very small amount of actual cellulose decomposition in terms of percentage loss in weight may bring about very major decreases in fiber strength.

Cuprammonium fluidity increases in weathered cotton fibers are frequently very large [31, 36, 42, 129]. All the available evidence indicates, however, that such fluidity changes are not due to microbiological but rather to nonbiological deterioration. Changes in fluidity accompanying extensive microbiological attack on cotton fabric are reported by Searle [111] to be insignificant or absent and by Rogers, Wheeler, and Humfeld [108] to be very low, not exceeding 10 percent of the original value after extreme tendering by *Chaetomium globosum*. Three months' sun exposure of a cotton fabric, on the other hand, was found in the experiments of Thaysen *et al.* [129] to result in viscosity decreases equivalent to approximately a half to two-thirds of the original values, while Fargher [42] reports even greater

viscosity decreases during the same interval of sun exposure; the two reports agree on the point that changes in viscosity are distinctly lower for fabric in shade exposure than for sun-exposed specimens. In confirmation of previous evidence, mentioned just above, Barghoorn [9], reporting on experimental results obtained by Rutherford, shows that severe tensile-strength losses of cotton under the influence of *Chaetomium globosum* are not accompanied by measurable changes in cuprammonium fluidity; sun exposure resulted in very large fluidity increases. Dean and Worner [36] working with fabric highly tendered by *Myrothecium verrucaria* (*Metarrhizium glutinosum*) found cuprammonium fluidity to be essentially unchanged by microbiological action. According to Nodder [91], weathered cotton fabrics damaged by light show large increases in sodium hydroxide solubility number, whereas fungus-damaged fibers show no such change.

Methods of Diagnosing Fiber and Textile Deterioration

Of the various methods which might be used in detecting and diagnosing microbiological fiber and textile deterioration, the most attention has been paid to certain microscopic techniques. Fleming and Thaysen [46] found that when damaged cotton fibers are subjected to the carbon disulfide-sodium hydroxide swelling technique originally described by Balls [8] they show a decreased tendency toward the beading phenomenon which occurs so strikingly with normal fibers. A similar observation had been made previously by Carbone [25] on microbiologically damaged fibers swollen in Schweitzer's reagent. Fibers damaged by microorganisms are also stated by Fleming and Thaysen to exhibit numerous cracks and incisions when tested by their method. Denham [37] criticizes the swelling technique of Fleming and Thaysen [46] as a diagnostic method for indicating microbiological damage on the ground that the reaction is dependent only on the behavior of the cuticle and that "slight mechanical damage to the cuticle, such as occurs in ginning, will produce fallacious results, while an organism which does not affect the cuticle, however extensive its ravages in the lumen and the wall layers, may escape observation." According to Bright [22], Thaysen in later conversation placed emphasis on the appearance of cracks and crevices in the swollen fibers as well as on the presence or absence of the beading phenomenon. He has himself [22] described another microscopic method for detecting fiber damage to cotton. His method again is apparently essentially a measure of the condition of the primary wall. It involves treatment of the fibers with 11-percent sodium hydroxide, then with a Congo red solution, and finally with 18-percent sodium hydroxide. The first, milder swelling is intended to rupture the primary wall of damaged but not of undamaged fibers, subsequent treatment with Congo red staining the underlying cellulose at these ruptured points. Final swelling in 18-percent sodium hydroxide brings about more extensive rupture of the cuticle and exposure of unstained cellulose. Clegg [29] presents several photomicrographs showing the results of tests on variously damaged cotton fibers by use of the Congo red test. Prindle [99] has used a combination swelling-staining technique with cuprammonium solution and Victoria blue stain for detection of mildew damage to cotton fibers.

Techniques described for staining fungus mycelium in cotton fibers include the use of cotton blue [53] and of basic fuchsin [108]. Bromphenol blue [110] and Pianese IIIb [76], the latter a mixture of Martius yellow, malachite green, and acid fuchsin, have also been used for examination of fungus-damaged fibers. Denham [37] presents a number of photomicrographs of deteriorated cotton fibers illustrating various types and stages of attack by bacteria and fungi. In some cases growth occurs down the lumen of the fiber and in other cases on the fiber surface. Stanier [116] illustrates his paper with interesting photomicrographs showing the corroding action of the cytophagas on the outer surface of cotton fibers; the pattern of the corrosion follows the spirals of cellulose structure in the fiber.

Fluidity determinations have frequently been used to indicate the extent of deterioration of weather-exposed cotton fabric [9, 36, 42, 111]; it appears that nonbiological rather than microbiological deterioration is reflected in results obtained by this method. Nodder [91] recommends the use of an alkali solubility test in distinguishing between light and microbiological damage in weathered cotton fabrics, high alkali solubility being characteristic of light-damaged fiber.

Strength determinations are often used in the diagnosis and detection of textile damage. Zuck and Diehl [142] devised a burst-strength tester for differentiating microbiological from nonbiological damage to fabrics. The distinction is made on the basis that fungus growth is commonly found in localized spots

and that by the use of this method strength tests may be made in fungus-spotted as compared with unspotted areas of the fabric.

Methods of Preventing Microbiological Fiber Deterioration

Use of Textiles of Inherently Low Sensitivity to Microbiological Attack

Fargher [42] discusses at some length the matter of "mildew capacity" of cotton and other natural fibers, a factor of particular importance in the mildewing of fabrics in storage and in transit. A large part of the information he presents on this point deals with the differences in the tendency to develop mildew in high-humidity exposure of different raw cottons and the general high susceptibility of raw cotton as contrasted with scoured and bleached cotton. This mildew-favoring property of unprocessed cotton or gray goods may be ascribed to any of several fungus-nutrition factors. All fungi are believed to have certain nitrogen and mineral requirements for growth and most of them utilize readily any of several sugars; these growth-supporting materials occur in different amounts in different lots of cotton fiber and may be largely removed during scouring. Vitamins, especially vitamins B_1, B_6, and biotin, are known to be required singly or in different combinations for the growth of many fungi. These vitamins occur in raw cotton [84, 106] but would also presumably be largely removed during scouring. The closely related [140] and very common cotton fabric destroyers, Memnoniella and Stachybotrys, have both been proved in the author's laboratory to require the vitamin biotin for their growth [84]. Many noncellulose-decomposing fungi can grow at the expense of the noncellulosic constituents present in gray goods but not in scoured goods and it seems not improbable that many cellulose destroyers grow first on the noncellulosic constituents before attacking the cellulose.

In out-of-door weathering experiments in the summer at New Orleans, Dean and Worner [36] found the visible appearance of mildew on the under surface of cotton sheeting to be much more pronounced on gray than on kier-boiled specimens. Year-round tests of gray as compared with kier-boiled and bleached print cloth, however, indicated that under New Orleans weather conditions the gray goods lost strength somewhat less rapidly than the kier-boiled. These results, suggesting the dominance of nonbiological as compared with microbiological factors of weathering with a lightweight fabric, may be contrasted with the dominance of microbiological deterioration of heavier fabric in the experiments of Zuck and Diehl [142], who found marked weakening of fungus-spotted as compared with unspotted areas in Florida-exposed duck.

The mildew capacity of fabrics is obviously increased by the use of any additives which support rapid fungus growth. Starch sizing has long been recognized to fall in this category, and different starches have been studied with respect to their tendency to support growth of mildew [88, 89]. Castor oil and various esters of stearic, oleic, and sebacic acids which may be used as plasticizers in textile coatings are known to stimulate growth of fungi. In tests carried out in the author's laboratory it was found, for example, that samples of dibutyl sebacate, butyl stearate, cottonseed oil, glyceryl monoricinoleate, methoxy ethyl oleate, and castor oil supported the growth of *Aspergillus niger*. Plasticizing agents may in some cases constitute a high percentage of a textile coating composition.

Certain fibers are completely resistant to rotting, being inert toward the digestive and deteriorative action of fungal and bacterial enzymes. Cellulose acetate fabrics have repeatedly been shown to resist microbiological action [21, 39, 65, 87, 127, 129].

Goldthwait, McLaren, and Voorhies [58] have prepared and reported results of microbiological tests on acetylated cotton—yarn, thread, and cloth—with acetyl contents ranging from 8 to 30 percent. Fabric of the lowest acetyl content, 8 percent, resisted attack in pure-culture tests by *Chaetomium globosum* and *Myrothecium verrucaria* (*Metarrhizium glutinosum*). Soil-burial tests indicated, however, that much higher acetyl contents are required for protection in soils. Fabrics containing 19, 25, and 30 percent acetyl showed 40, 11, and 2 percent strength losses, respectively, after 5 weeks, and 100, 52, and 9 percent strength losses after 50 weeks in soil. Sandbags made from cotton cloth and thread of high acetyl content were piled in stacks on the ground and were found to remain intact after 2 full years of exposure in New Orleans.

"Ban-Flame," a fabric flameproofing finish, represents a chemically modified cellulose, believed to be a cellulose phosphate [9]; samples tested in the writer's laboratory have proved inert to fungi and entirely resistant to degradation in soil-burial tests. Barghoorn [9] found a sample of fabric which had received the same treatment to be very susceptible to

tendering in sunlight. Siu and White [115] have reported preliminary results obtained in a study of the resistance toward microbiological action of various cellulose derivatives. According to their experiments even minor changes in cellulose in the nature of chemical substitutions may bring about resistance or inertness toward cellulose-decomposing fungi.

Nylon and Vinyon are inert toward the action of fungi [87] and glass fiber is, of course, not attacked. The U. S. Army Engineer Corps combated the problem of rotting of portable fabric water reservoirs during World War II by the use of rubber-coated glass fabric. Glass fiber was considered to have an advantage over cotton for use in braid on electrical wiring during the war because of its inertness to mildew. In some cases, as, for example, in the lighting of air fields, electrical cable lines were buried under ground, a severe condition conducive to rapid rotting.

Although certain plant fibers other than cotton have been stated in various popular and semi-popular articles to be highly resistant to rotting, very little factual data appear to have been published on the subject. Lignin is believed by some to increase the resistance of cellulosic fibers to attack by microorganisms. Coir fiber, obtained from the husk of the coconut and containing as much as 30 percent lignin, is said to be highly resistant to microbiological decomposition [94]. It is used in tropical Asia and the South Seas in the manufacture of hawsers, cables, and small cordage [66, p. 44]. Jute, however, containing on the average about 12 percent lignin, is rapidly deteriorated by *Chaetomium globosum* and has little resistance to rotting in soil. Abaca and ramie are said by Dewey [38] to show high resistance to "sea water." Claims for great rot-resistance have appeared in several popular articles on ramie but have not, to the writer's knowledge, been supported by published data.

The general conception prevails among workers concerned with fiber deterioration that under most circumstances wool is less susceptible to microbiological degradation than many of the natural plant fibers. Galloway and Burgess [56] offer this comment: "Wool and other hair fibers differ from the vegetable fibers in being essentially protein and not cellulosic in nature. Under ordinary conditions they are probably less susceptible than vegetable fibers to fungal attack, but are more susceptible to certain bacteria." The susceptibility of wool to bacterial deterioration is of practical importance in the case of woolen paper-mill felts, which are commonly exposed to conditions highly favorable to bacterial growth and are severely attacked in some instances [20]. Waters [135] has described a condition known as "pink rot," found to occur in wool on sheep's backs. A bacterium isolated from the deteriorated wool and resembling *Bacillus vulgatus* in its cultural reactions was found to produce rapid deterioration in the laboratory. Photographs showing progressive degrees of damage to a wool fiber under the influence of bacteria are presented in a paper by Bartsch [14]. He states that *Bacillus subtilis* and *Bacillus mesentericus* are the most destructive. The biological attack appears to take place primarily on the cementing substances holding together the spindle-shaped cells within the fiber. Wool is more readily attacked by bacteria if it has been swollen previously in alkali or in boiling water [15]. Autoclaving of wet wool under the conditions customarily used in pure-culture work with microorganisms causes chemical degradation [72] and probably increases the fibers' susceptibility to subsequent biological attack. It is generally believed that natural silk is relatively resistant to microbiological attack [15].

Moisture Control

The moisture content of a fiber and of the atmosphere surrounding it has a pronounced influence on the growth of mildew. Armstead and Harland [4], who are quoted frequently on the point, give the figure 7.5 percent moisture as the limit below which no fungus growth will be observed on cotton fabrics. In the opinion of Fleming and Thaysen [45] the minimum moisture content which will allow organisms to develop on raw cotton is about 9 percent. Prindle [100] incubated cotton at different relative humidities and observed a distinct moldy odor at values of 85–95 percent relative humidity (86 percent corresponds to about 12 percent moisture content); mildewing at 82 percent relative humidity (10.5 percent moisture content) was not so pronounced as that at the higher humidities. According to Galloway [55] 15 percent moisture regain approximates the upper limit for safety in storing jute. Finlow [44] claims that "heart damage" to jute—microbiological deterioration at the center of the bale—is in fact due to improper drying or to "watering" of the fiber by exposure to night dew, etc. During the recent war emphasis was placed on proper design of storage fa-

cilities to insure freedom of air movement and to avoid high humidity.

Use of Mildew Preventives

Perhaps the oldest and best known of the various compounds which have been employed to protect textile fibers against biological degradation is copper naphthenate. Used for many years in Europe as a preservative for fish nets [7, 33], it was recommended among other compounds by Armstrong [5, 6] in 1941 for rotproofing sandbags in England, and was subsequently utilized on a large scale both there [33] and in the United States [57] for that purpose. Armstrong [6] states, without offering definite experimental evidence: "The very best results have been obtained by proofing with a combination of creosote and copper naphthenate in which the tar compound besides being toxic exerts a waterproofing effect in delaying the loss of copper by leaching." Actually, Armstrong's contention regarding the superior performance of the copper naphthenate-creosote combination on sandbags is borne out by the results of simulated service-condition exposure tests of sandbags as described by Dean, Strickland, and Berard [35]. Copper naphthenate may be applied to fabric as a solution or suspension in organic solvent or ammonia, application from organic solvents having been to date the most common method. The material was used extensively by the U. S. Army in the latter part of World War II in combination with hydroxy copper naphthenate as the mildew-preventive component of a treating compound for preparing fire-, water-, and weather-resistant tent canvas [28, 80–2], and was employed also in the mildewproofing of camouflage netting, webbing, thread [23], and cordage [131, 132]. Cave-Browne-Cave and Clark [27] carried out experiments with copper naphthenate in combination with chlorinated rubber and various plasticizers to increase the life of fish nets. While preliminary tests on sections of twine indicated an increase in durability in sea water, subsequent trials with actual fish nets were not entirely satisfactory in that the preservative did not sufficiently penetrate the knots and protection was consequently inadequate.

The effectiveness of copper naphthenate in pure-culture, soil-burial, and soil-suspension fabric tests has been reported by various authors [2, 16, 17, 34, 50, 51, 86, 87, 131, 132]. Although "naphthenic acid" is a complex mixture of substances which undoubtedly varies in composition from one source to another, it has been common experience among workers in the field of fabric mildew preventives to find that copper naphthenates from various sources possess unusual fungicidal properties not common to most other copper compounds. Experimental work in the author's laboratory has consistently revealed this superiority [86, 87], which, it is believed, may be attributed to fungicidal activity in the naphthenate component of the material [86]. This conclusion is already supported by data of several kinds [86] and it may be mentioned here as further evidence that a sample of nickel naphthenate showed distinctly greater fungicidal effectiveness in a soil-burial experiment in four different soils in the writer's laboratory than did one of nickel oleate, metal concentrations of 0.1, 0.2, 0.4, and 0.8 percents on an 8-ounce cotton duck allowing only 93, 40, 19, and 3 average percents of strength loss for the former compound as compared with 100, 100, 100, and 98 percents for the latter.

Although copper naphthenate seems to have better retention on fabric than certain of the pure organic compounds when tested by water-leaching procedures in the laboratory [87], in out-of-door exposure the copper content of the fabric decreases rather rapidly [5, 6, 42]. Bayley and Weatherburn find that a wax coating may exert a very pronounced protective effect toward the loss of copper on weathering from either fabric [17] or cordage fibers [18]. The same authors [17] report, however, that water-leaching of certain copper-naphthenate-treated fabrics in their experiments, although accompanied by only minor losses of copper from the fabric, brought about marked decreases in resistance to strength loss in subsequent soil-burial tests. The effect was much less marked or even absent when the copper naphthenate was present in association with a waterproofing wax. It was apparently associated with a change from solvent-soluble to solvent-insoluble copper.

The question has arisen from time to time of whether or not copper naphthenate may stimulate the nonbiological tendering of textiles during weather exposure. The most thorough study of this question published to date appears to be that made by Bayley and Weatherburn [17–19]. According to their results, copper naphthenate has no stimulative effect on nonbiological weather damage to fibers. In the case of treatments applied without wax the absence of a deteriorative effect might possibly have been associated with rapid loss of copper from the fabric, such major losses being shown by the authors' data.

In the case of copper naphthenate treatments applied with wax, however, the losses of copper were low and still no copper-naphthenate-stimulated deterioration was observed. Bartlett and Goll [13] had previously concluded that copper naphthenate does not stimulate nonbiological tendering of cotton or jute, on the basis of data from short-time laboratory exposures of copper-naphthenate-treated fabric to ultraviolet light at elevated temperature in the presence of oxygen and from 4½ months' weather exposure tests at Elizabeth, New Jersey. Barghoorn [9], on the other hand, obtained distinct stimulation of light tendering with copper naphthenate on cotton sheeting in his experiments. Unfortunately, in Barghoorn's experiments [9, p. 23–24] the fungicidally treated fabric samples differed from the unmildewproofed samples in that there was present a water-repellent as well as a fungicide. It is impossible, therefore, to attribute the stimulative effect on fiber deterioration to the fungicide rather than to the water-repellent or to be certain that any effect of the fungicide was not influenced by the water-repellent or even by the dye treatment which was present on both mildewproofed and unmildewproofed samples. In experiments by Bayley and Weatherburn [19] a petrolatum-containing waterproofing wax brought about decided acceleration of nonbiological deterioration of cotton fabric on weather exposure. In view of the importance of copper naphthenate as a mildew preventive, further investigations regarding any possible influence of the material on nonbiological weather damage would seem to be desirable.

Of the several copper compounds other than copper naphthenate and hydroxy copper naphthenate which have been used as fabric preservatives, copper oleate, copper ammonium fluoride, copper "tallate," and cuprammonium are probably the best known. Copper oleate was used during the war in combination with a mercurial in certain fire-, water-, and weather-resistant tent-duck finishes; the other three were applied to sandbags. Copper resinate and copper oleate have been used for some time in the preservation of fish nets [107]. Of these several compounds, all have proved to have less fungicidal effect per unit weight of copper on fabric than copper naphthenate [35, 86, 87]. A cuprammonium-creosote emulsion treatment was found highly effective in preventing rotting of sandbags in the experiments of Dean, Strickland, and Berard [35].

Apparently none of the copper compounds has been shown to persist well on cotton fabric during weather exposure unless protected by a coating of wax or similar material. Jarrell, Stuart, and Holman [75] found certain inorganic copper treatments, both with and without mineral khaki dyeing, to be very fugitive during weathering. They treated fabric by two-bath procedures involving first impregnation with soluble salts of chromium (chromium-potassium sulfate or chromium acetate), iron (ferric sulfate or pyrolignite), or copper (copper acetate), or various combinations of these salts, followed by a sodium carbonate precipitation on the fiber. Most, and in some cases all, of the copper was lost from the fabric in 6 months of weather exposure, the chromium and iron appearing to have no tendency to improve copper retention. Fargher [42] reports essentially the same results, as do also Race and Rowe [102].

According to the results of Jarrell, Stuart, and Holman [75, table 2], copper deposited on cotton fabric by the use of copper acetate followed by sodium carbonate had a tendency to accentuate nonbiological tendering during exposure on weather racks. With an iron-copper treatment precipitated on a Florentine drill with sodium carbonate, Fargher [42] found distinct stimulation of nonbiological tendering; copper precipitated alone or applied as cuprammonium did not accentuate tendering.

Zinc naphthenate has been used on fabrics on which copper naphthenate or other copper compounds would be undesirable because of their color or because of their tendency to accelerate deterioration of coatings —for example, in rubberized pontoon fabric and in coated aircraft fabric. Zinc naphthenate is less fungicidally active than copper naphthenate but fungicidally superior to the corresponding zinc oleate, "tallate," or hydrogenated-resinate [86] exactly as copper naphthenate is fungicidally superior to these related copper soaps.

Several mercury compounds have been used as mildew preventives on fabric in this country, perhaps the best known of these being a material whose chemical composition is said to be phenyl mercury triethanolammonium lactate. The compound is applied in aqueous solution. Phenyl mercury oleate has also been used to a limited extent [59]. Pyridyl mercury stearate has been tested in several laboratories but apparently has had only limited commercial application. Many of the mercury compounds are highly effective against *Chaetomium globosum, Myrothecium verrucaria* (*Metarrhizium glutinosum*) and certain

other fungi in culture tests but are much less effective against certain Penicillium species and notably poor at the ordinary commercial concentrations in soil-contact exposures [87]. Mercurials (as well as other types of compounds) were used during the war in wire-coating compositions for moisture- and fungus-proofing electronic equipment [69]. The fungicide was intended to prevent growth of fungi on cotton braid-wire covering or other mildew-susceptible components of the equipment or on contaminating materials which might be introduced during service. The results obtained were not entirely satisfactory and further research on the subject is being carried out in various laboratories.

Fargher [42] has attributed a moderate mildew-preventive function to mineral khaki treatments on cotton fabric, presumably owing to the presence of chromium. Jarrell, Stuart, and Holman [75] were unable to find that such treatments afford protection against *Chaetomium globosum*. More recently, Race, Rowe, and Speakman [103] have studied the fungicidal properties of various mineral khaki treatments on cotton. They state: ". . . the only toxic principle in a chrome-iron-treated yarn is hexavalent chromium, and that when hexavalent chromium is absent from the pigment, the trivalent chromium and iron compounds impart to the yarn a resistance, but not a toxicity, towards bacteria and fungi. Therefore, since hexavalent chromium is produced only by the chromate development process, and since this hexavalent chromium is not permanent, but is hydrolyzed by water and reduced by the cellulose of the material and possibly by reducing products of metabolism, it follows that the proof imparted to cotton by this pigment is also not permanent."

Pentachlorophenol is a material which because of its extremely high fungicidal potency [87] and moderate price might be considered to offer good practical potentialities as a mildew preventive. It was, in fact, employed extensively during the early part of World War II for mildewproofing U. S. Army fire-, water-, and weather-resistant tent canvas [73]. Its use for this purpose was subsequently discontinued, however, when it became evident that the material was quickly lost from the fabric during weathering. It was also employed on camouflage fabric. Unpublished data from this and other laboratories indicate that in out-of-door weathering pentachlorophenol may under some circumstances significantly accelerate nonbiological tendering of fabric. The exact circumstances under which such stimulation takes place are not known to the author to have been adequately defined in any published work. According to the experimental results of Barghoorn [9], stimulation of nonbiological tendering appears to be a common characteristic of a wide variety of mildew-preventive agents, particularly in the presence of light. As has been previously noted, however (p. 605), certain difficulties are encountered in the interpretation of Barghoorn's data. The retention of pentachlorophenol on cotton fabrics exposed to water-leaching is notably poor. Under some conditions the compound is highly irritating to the skin.

2,2′-methylenebis(4-chlorophenol), also referred to as 2,2′-dihydroxy-5,5′-dichloro diphenylmethane, was marketed under the name of "Compound G-4" or "Preventol GD" during the latter part of World War II and was used on a large scale as a mildew preventive on various U. S. Army fabric items such as dyed duck, insect netting, and webbing. It is a white powder, with little odor, and with apparently little or no irritant effect on the skin. Its unusual fungicidal effectiveness has been demonstrated against fungi in pure culture and in soil-contact fabric tests [2, 85, 87]. Unfortunately, however, like pentachlorophenol, the compound is not well retained on fabric during laboratory water-leaching [87] and appears to be lost readily from fabric during weathering [19].

A communication from the author's laboratory [85] deals with the fungicidal activity of several bisphenols related in chemical structure to Compound G-4. It has been found that a number of these have high fungicidal activity and that certain rather clear-cut relationships exist between chemical structure and fungicidal activity within the group, compounds most closely resembling Compound G-4 in structure having the highest activity. For a review of earlier information on the relation between chemical structure and microbiological activity among the phenols, the reader is referred to a paper by Suter [126].

Salicylanilide ("Shirlan") was originally recommended by Fargher, Galloway, and Probert [43] as a material to be incorporated into sizing to prevent mold growth on fabric during storage and transport, and has been widely used for that purpose. It has been found to be distinctly more effective than zinc chloride, which was used earlier for the same purpose. Fargher [42] does not recommend salicylanilide for use on fabric to be exposed to weathering in

service, presumably because of its poor adherence under these conditions.

Furry [49] studied the mildewproofing effectiveness of several natural dye-copper sulfate-potassium dichromate treatments. All of the dyes were tannin-containing materials applied in the presence of copper sulfate and fixed on the fabric by a boiling solution of potassium dichromate. Several of the treatments were effective in a soil-suspension test.

A large number of organic and metal-organic compounds other than those mentioned above have been recommended by one author or another as fabric mildew preventives without ever having come into common practical use for that purpose. 2,3-dichloro-1,4-naphthoquinone was recommended by ter Horst and Felix [71]. Copper pentachlorophenate, the copper salt of 8-hydroxyquinoline, zinc dimethyl dithiocarbamate, mercaptobenzothiazole, phenyl mercury acetate—these and many others have been investigated in laboratory studies but have apparently not been extensively used in practice. A cadmium soap treatment effective against *Chaetomium globosum* was described by Furry *et al.* [50]. Fargher, Galloway, and Probert [43] mention the high fungicidal activity of thallium carbonate, *o*-chloromercuriphenol, *p*-acetoxymercuriacetanilide, *p*-nitrophenol, and trichlorophenol but conclude that none of these is practical for incorporation into sizing to prevent mold growth. Recent progress in the field of new fungicidal compounds has been reviewed by Horsfall [70].

Preliminary experiments with cotton seine twine by the Bureau of Agricultural and Industrial Chemistry of the U. S. Department of Agriculture [26, p. 13] have employed the method of textile impregnation with furfuryl alcohol followed by resinification of this unsaturated compound by polymerization within and on the fibers; results of these preliminary tests are said to have been promising. The principle of physically protecting a fiber against biological attack by coating or impregnating it with material inert toward microbiological action has attracted the attention of various investigators but the method has generally failed because of discontinuity, impermanence, or other undesirable properties of the protective material.

Testing Fabrics for Behavior Toward Fungi

Certain relatively simple tests can be performed in the laboratory which, although not taking the place of the more time-consuming actual service-exposure trials, may in many cases be expected, upon careful interpretation, to yield useful information regarding the behavior toward mildew of textiles or textile mildew preventives. Most experience has been obtained with fabrics and fabric mildew preventives. While the reader must be referred to original articles and papers for detailed information on the subject [2, 3, 11, 34, 51, 62, 87, 130], an attempt will be made here to summarize the essential facts and concepts which are developed at greater length in these publications.

Pure-Culture Tests

Although a number of different fungi have been found to offer promise as fabric mildew-resistance test organisms in various laboratories, it is probably accurate to say that none of them has as yet been shown to have general superiority over the *Chaetomium globosum* isolate 1042.4 originally recommended for this purpose by Thom, Humfeld, and Holman in 1934 [130]. The organism combines a number of suitable characteristics. It deteriorates cotton, jute, and other cellulosic fibers rapidly, is very easily maintained in pure culture, and has shown no tendency to be unstable in its properties. Much data has already been accumulated with *Chaetomium globosum* 1042.4; this fact in itself is a point in favor of its continued use, since in this way test results with new mildew preventives or textiles may be compared with results on many other previously tested preventive compounds and textiles. Under certain circumstances it may be desirable to test mildew-preventive compounds on fabrics or other textiles without sterilization in order to avoid the influence of steam on the mildew preventive. While no test fungus can be recommended without reservation at the present time for use in nonsterile techniques, *Chaetomium globosum* 1042.4 has been found in the writer's laboratory to be more reliable in its performance than any of several other fungi for use under these conditions and has been used in this fashion in many tests [85, 87].

A technique which has been found by experience to be very satisfactory in conducting *Chaetomium globosum* mildew-resistance tests on fabric consists in raveling 6-by-1½-inch test strips to 1 inch in width, sterilizing them by steam at 15 pounds pressure for 15 minutes, planting the sterilized strips on mineral salts-agar in 16-ounce flat-sided bottles, inoculating each strip with 1 cc. of a suspension of

spores of the fungus, incubating the cultures at 30°C for 12–14 days, and then determining the residual strength of the fabric. Various kinds and quantities of mineral salts may be used in the medium; of the several compositions which have been tried by various workers, the following is apparently as satisfactory as any:

NH_4NO_3	3.0 g.
K_2HPO_4	2.1 g.
KH_2PO_4	2.7 g.
$MgSO_4$-$7H_2O$	2.2 g.
Water	to 1,000 cc.
Agar	20 g.

This medium is known to be well adapted to the growth of many cellulose-decomposing fungi in a test procedure identical in all respects with the *Chaetomium globosum* technique just described. The medium may be varied in composition over rather wide limits and still provide good growth and fabric breakdown as long as it contains a sufficient quantity of the elements essential for fungus growth—namely, nitrogen, phosphorus, potassium, sulfur, and magnesium. The present writer is of the opinion that the higher nitrogen requirement of *Chaetomium globosum* in the experiments of Romano [109], as compared with the earlier results of Greathouse, Klemme, and Barker [62], was in actuality a result of the incompleteness of diffusion of salts to the middle of the test strips in Romano's experiments, this interpretation being suggested by the marginal nature of the fungus growth as seen in the photographs in his paper. For general information on the inorganic nutrition of fungi, the reader is referred to a review by Steinberg [121].

Since a high-percent strength loss is readily obtained with fabric as heavy as 8-ounce duck in tests with *Chaetomium globosum* 1042.4 in tightly closed 16-ounce culture bottles [87], it is probably not necessary in such tests to use special glass-cloth aeration vents for culture bottle tops [62] unless, for some special reason, complete strength loss is desired.

Fungus isolate 1334.2, known until recently as *Metarrhizium glutinosum* [2, 62, 86, 98], is extremely rapid in its tendering action on cotton and other natural fibers in laboratory tests and is very readily handled in culture work. Experience suggests that it is probably very stable in its properties. Recent observations by White and Downing [139] have shown that the organism is in reality not a Metarrhizium but rather a strain of the previously described *Myrothecium verrucaria* and should properly be called by that name. These authors [139] remark upon the widespread use made of this fungus in fabric testing, a situation which they apparently find hard to reconcile with their own observations, which lead them to state that "Despite its strong cellulolytic action in pure culture it is considered to be of little or no importance in the decomposition of cotton fabrics under field conditions." Speed was, of course, one of the prime requirements of fabric testing for military procurement during the war and tests during the early period of the war had shown that "Metarrhizium" was in fact very similar in its behavior toward mildew preventives to the slower-growing *Chaetomium globosum*. The writer, however, has also been of the opinion for some time that "Metarrhizium" is of relatively infrequent occurrence on deteriorated fabrics. The fungus was referred to in a publication in 1944 as an "extraordinarily active" cellulose decomposer in contrast to Chaetomium, which was termed a "common" decomposer [86, p. 177], and its infrequence of occurrence, together with the several positive merits of *Chaetomium globosum*, accounts for the appearance of results of some 1,320 tests with the latter organism as compared with only 115 for "Metarrhizium" in a 1945 publication on test methods [87].

In such cases as it might be desired to carry out fabric tests with cellulose-decomposers other than *Chaetomium globosum* 1042.4 or *Myrothecium verrucaria* 1334.2, it will be found that many fungi will produce rapid fabric tendering when used in the manner just described in the sterile Chaetomium procedure. Some of these organisms are distinctly more tolerant toward fungicides than either Chaetomium or Myrothecium, especially toward those containing copper or mercury.

Superficial as well as cellulose-decomposing fungi have been observed to grow on textile products under service conditions—for example, on water-repellent waxes, fabrics coated with compositions containing certain plasticizing agents, etc. It was common practice during World War II to test textile articles for resistance to growth of superficial as well as cellulose-decomposing fungi. The most common organism used for this purpose was *Aspergillus niger*, in particular an isolate designated as TC-215-4247. The nutritional requirements of this fungus have been studied in detail by Steinberg (117–125) and others

and are probably as well if not better known than those of any other fungus. Isolate TC-215-4247 is the same single-spore isolate used by Steinberg in his investigations. The fungus utilizes readily either nitrate or ammonium nitrogen. In addition to the usual macro elements—potassium, magnesium, sulfur, and phosphorus—it requires extremely minute amounts of iron, zinc, manganese, copper, and, under special conditions, molybdenum. The amounts of trace elements required are, however, very low; in his dibasal solutions Steinberg [125] used each of the required trace elements at 0.02 to 0.4 mg. per liter. It is generally assumed, although the point is admittedly a debatable one, that under the conditions of fabric mildew-resistance tests it is unnecessary to add trace elements to the medium since they will be introduced in adequate amounts in the test specimen as well as in the form of impurities in the salts and agar of the test medium.

Aspergillus niger is used in solving two basically different problems: (*a*) to determine the relative fungicidal activity of mildew preventives and (*b*) to determine the mildew-response behavior of noncellulosic textile additives or noncellulosic fibers which may serve as food sources for fungus growth or which may be physically or chemically altered by microorganisms.

Soil-Contact Tests

"Soil burial" tests are commonly carried out by planting in soil small samples of the fabric to be tested, allowing 1 to 4, or in some cases more weeks for incubation, and testing the residual strength after burial [34, 87]. It is customary practice to carry out the tests indoors in temperature-controlled rooms, the soil being placed in covered boxes, aquaria, or other vessels for the test. The best soils for the purpose are mineral soils intermediate in clay and organic-matter content, of the type which would be recognized as good garden loams.

While soil-burial tests were formerly thought by various investigators, particularly prior to about 1943, to be very unsatisfactory because of poor reproducibility of results obtained, the method's reputation appears to have improved materially in the minds of many workers, experience having shown that proper control of temperature and soil moisture will eliminate a great deal of the highly erratic results characteristic of early tests by this method. A temperature of 85°F has been found to be generally satisfactory; a moderate soil moisture level should be maintained, either very dry or very wet conditions being undesirable.

While the absolute figures for results in soil-burial tests are seldom reproducible with extreme accuracy from one test to the next and between laboratories, the relative rankings of different fabrics and mildew preventives are commonly in at least approximately the same order in different soils and in different tests in the same soil and the differences obtained among different compounds and treatments are frequently very large so that certain general conclusions may be drawn from the data obtained. Two specific instances may be cited. The behavior of copper naphthenate in different soils in this [86, 87], and in other laboratories [17], has been consistently superior to that of several other copper preservatives. Mercury compounds, in the customary commercial concentrations applied to fabrics during recent years, have been very generally found inadequate for protection in soils [for example, 2, 87]. A large number of compounds of mediocre-to-average fungicidal activity have been consistently and definitely eliminated by the soil-burial test procedure from consideration for use on fabrics to be exposed to soils in service.

In 1943, Furry and Zametkin [51] introduced a fabric rot-resistance test under the designation of a "soil suspension" method. In this procedure the strips are inoculated with a variety of organisms by dipping into a thin soil suspension; they are subsequently incubated in culture bottles on a mineral salts medium and the test results are evaluated in terms of residual strength.

Out-of-Door Weathering Tests

These tests are usually carried out by simply exposing fabric samples of any suitable size to the weather and determining their breaking strengths after various periods of exposure on weathering racks. The tests may be performed either in the sun or in the shade; in the former case the racks are commonly arranged to face south with the samples held in a position inclined 45° to the horizontal [36]. Exposures may be continued for periods of from a few weeks to as long as 3 or 4 years. In case a fabric is very poor in its resistance to nonbiological tendering this fact may be readily apparent from results of tests lasting only 4 or 5 weeks; this is particularly true if the fabric is light in weight.

Studies of the behavior of mildewproofed fabrics

during exposure to the elements on weathering racks have been made by English workers [42] and in this country at the Southern Regional Research Laboratory and by various divisions of the Armed Forces [9]. Much of the data obtained in this country have not been published; the conclusions reached, however, are believed to be similar in certain respects to those presented by Fargher [42]—namely, that most compounds adhere very poorly to cotton fabric in weather exposure and that some of them stimulate fabric tendering. Fynn and Campbell [52] have described an automatic normal-incidence solar exposure cabinet for experimental work relating to the effect of sunlight on fabrics.

"Tropical Chambers"

Cooke and Vicklund [32] have reported on the construction of a large "tropical chamber" used by the U. S. Army Engineer Corps at Fort Belvoir, Virginia, in studying the behavior with respect to mildew and high humidity of many finished articles of Army equipment. Their chamber has the advantage of being able to accommodate large items; it is adequate in size, for example, to hold a large truck. Less elaborate chambers ranging from temperature-controlled rooms with moisture introduced through atomizers or as slow-leaking steam, down to moist chambers in the form of tightly covered aquaria, desiccators, and bottles of various sorts have been used in other places. In moist atmosphere exposures much the best fungus growth appears to take place on textiles at or very close to 100 percent relative humidity; humidities of 95 percent and below are much less satisfactory.

Chemical Analyses

Correct interpretation of the results of fabric mildew-resistance tests is frequently entirely dependent on a knowledge of the exact amount of a mildew-preventive agent on the fabric. While details of applicable analytical procedures are in some cases available in textbooks, in other cases suitable methods are found only in recent publications or are in the hands of manufacturers of mildew preventives. The standard iodometric method for the determination of copper as described by Kolthoff and Sandell [79], as well as the standard electrolytic methods, has been commonly used for the determination of copper on fabrics. Weatherburn, Weatherburn, and Bayley [136] have presented a method especially adapted to determining copper in copper naphthenate. Shiraeff [113] describes a dithizone method for estimating mercury in mercury-organic mildew preventives on fabrics. In one method for its quantitative chemical determination pentachlorophenol is converted into an orange-colored compound in the presence of hot 2–1 nitric acid and benzene; the colored benzene layer is dried with anhydrous sodium sulfate and the color is measured in a photoelectric colorimeter. The colorimetric reaction of phenols with 4-amino antipyrine, originally described by Emerson [41], has been adapted to the quantitative determination of "Compound G-4" and other phenols [60]. Salicylanilide produces a color in the presence of diazotized *p*-nitroaniline-*o*-sulfonic acid which may be used for colorimetric estimation.

Summary

Deterioration of textiles may be caused by either biological or nonbiological agents. Either type of deterioration may be severe, depending on the conditions of exposure.

Among the biological agents which cause textile deterioration, most attention has been paid to the fungi, many of which are known to be active cellulose decomposers and to bring about rapid weakening of cotton and other natural or regenerated cellulosic fibers. The importance of bacteria has not been adequately investigated although it seems probable from prior knowledge of their high moisture requirements for growth that except in the case of fiber deterioration in aquatic exposures or under other conditions involving the continued presence of actual liquid water films on the fibers they are less important than fungi. Wool is probably more susceptible to bacteria than to fungi. Nonbiological deterioration is believed to be due in many cases to photochemical degradation of fibers in sunlight.

The effects of microbiological deterioration on fibers and fabrics vary with the conditions and lengths of the exposure. Under high-humidity storage conditions deterioration may be evidenced by a musty odor and darkening of the fiber, sometimes accompanied by visible fungus fruiting structures and resulting ultimately in decreases in fiber strength and in some cases in increased brittleness of the fiber. In above-ground exposures out-of-doors fungus growth on fabrics is often apparent within a few days to a few weeks, developing with time into isolated dark spots or larger dark areas which in moderate-to-

high light exposures occur predominantly on the under side of the fabric. In soil contact the fiber is frequently reduced to zero strength and in many cases is completely digested on very short exposure. Evidence presented to date suggests that changes in cuprammonium fluidity, observed in marked degree with some weathered cotton fabrics, are due to nonbiological rather than to microbiological deterioration.

Improvements are needed in methods for detecting and diagnosing fiber and fabric deterioration, particularly in the incipient stages. Two microscopic tests have been used for this purpose—namely, the Fleming-Thaysen test and the Bright test. The former depends on the swelling behavior of fibers in sodium hydroxide-carbon disulfide and the latter on the staining properties of alkali-swollen fibers exposed to Congo red.

Three different general methods of preventing biological deterioration of fabrics and other textiles have been suggested, each claimed by certain investigators to have proved applicable in practice to certain rather specific situations. (1) Use of textiles of inherently low sensitivity to biological attack is effective in some instances. In the case of cotton fabrics exposed to high humidity but not to extensive contamination with foreign substances during transit and storage, it has been observed that scouring to reduce the content of noncellulosic impurities on the fiber has a marked effect in reducing mildew. Starch and certain other fabric additives tend to have the opposite effect. Fibers made from certain materials, as, for example, cellulose acetate and nylon, are inert toward the deteriorative action of microorganisms, which do not possess enzymes adapted to the decomposition of such substances. (2) Maintenance of low-to-moderate levels of atmospheric humidity is a practical control measure under certain circumstances. (3) Mildew preventives, particularly for fabrics, have been used on an extensive scale, particularly during the recent war, and have proved beneficial in certain cases.

Laboratory mildew-resistance test methods have been devised which, although not taking the place of longer service-exposure trials, have in practice proved useful for specific purposes. It is possible by their proper application to determine the relative fungicidal potency of different mildew preventives, the effectiveness or noneffectiveness of a fungicidal treatment on a fabric or other textile at any time before or during its service life in preventing growth of fungi, and the inherent ability of a fiber to serve as a source of food to fungi or to be tendered or otherwise affected by them. When such microbiological tests are used in combination with out-of-door exposures, it is possible to determine in a general way the persistence of mildew preventives on textiles; chemical analytical methods are available in some cases to accomplish the same purpose.

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Studies of the Treatment of Cotton in Sliver Form

II. Effect of Syton: Spinning Studies*

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THE first paper in this series [1] described the development of an apparatus for the continuous treatment of cotton fibers in sliver form and the effect of colloidal silica (Syton W-20) in the spinning of fibers treated in the card sliver state. It was found that the twist required for the development of maximum strength was reduced markedly below that required for untreated cotton samples in all cases investigated. This paper is devoted to studies of the treatment of drawing sliver and of yarn, and the effects of temperature and humidity variations under which spinning of the treated sliver was carried out.

Treatment of Drawing Sliver

Since the primary effect of the silica dispersion appeared to be an increase in interfiber friction, it seemed advisable to investigate the application of the sol to fibers in a more nearly oriented condition than is encountered in card sliver. In accordance with Balls' "second paradox" [2]—"Up to the front mule roller, cotton must be slippery; afterwards it must be sticky"—the ideal point of application of the silica is at the spinning frame, between the front roll and the bobbin. This approach was deferred, however, because of the mechanical details involved in a multiple-end treatment on such a machine as a spinning frame. An investigation of drawing sliver treatments was undertaken as the next step in this work, since only minor modifications in the technique previously described were necessary to permit the treatment of fibers in this form.

Crude experiments indicated that drawing sliver is roughly half as strong as card sliver of comparable weight, and breaks at an elongation of approximately one-half the amount realized in the breaking of card sliver. It was necessary, therefore, to reduce the draft in the treating apparatus to a minimum in the treatment of drawing sliver, but with some care in handling, the impregnation of drawing sliver did not offer unusual difficulties.

To compare the treatment of card and drawing slivers with respect to yarn strength, sufficient quantities of each were treated with identical Syton dispersions for spinning tests. The solutions contained 0.25 percent of Aerosol OT and 12.9 percent of Syton W-20 by weight, and had a total solids content of 2.37 percent. Add-on and spinning-test data appear in Table I.

The treated drawing sliver was more easily handled during the spinning than the treated card sliver processed in the preceding studies and produced a yarn of significantly higher strength at an identical silica concentration. Whether this difference was due to an increase in silica retention during spinning or to a more even drafting of the fibers in drawing sliver was not determined.

Processing Variables

Mechanical Loss of Silica

In all cases, the loss of silica from treated cotton during subsequent operations was evident, mainly

TABLE I. TREATMENT OF CARD AND DRAWING SLIVERS

Sample	Apparent add-on, weight percent of dry cotton	Corrected skein strength of 11s yarn (lbs.) Twist multiple 4.50	4.00	3.50
Untreated cotton	—	225	216	207
Card sliver	2.72	241	248	252
Drawing sliver	2.72	261	272	278

* The first paper in this series appeared in the June, 1947, issue, page 331.

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TABLE II. LOSS OF ASH DURING PROCESSING

Sample	Apparent add-on, weight percent of dry cotton	Ash content—weight percent of dry cotton: Clean impregnation bath: Card sliver	Drawing sliver	Roving	Yarn	Old impregnation bath: Card sliver	Drawing sliver	Roving	Yarn
Untreated cotton	—	1.12	—	—	—	—	—	—	—
1	0.47	0.43	0.53	0.49	0.51	0.98	0.97	0.98	0.95
2	0.72	0.52	0.60	0.59	0.60	1.16	1.26	1.22	1.25
3	1.08	1.31	0.80	0.70	0.69	1.52	1.47	1.48	1.41
4	2.06	1.22	1.18	1.15	1.14	2.37	2.19	2.12	2.14
5	2.89	1.75	1.63	1.63	1.45	2.83	2.56	2.51	2.46

through deposition of silica on the machines. Loss of silica appeared to depend somewhat on the concentration present in the treated sliver, and was more severe in the drawing operation than in roving or spinning.

To estimate the loss of silica during drawing, roving, and spinning, a series of identical card sliver samples were treated with dispersions of from 1 to 15 percent Syton W-20 and processed into 11s yarn in the usual way. Aerosol OT in 0.25-percent concentration was used as a wetting agent in all instances. Loss in ash content of the treated cotton during processing was chosen as a measure of silica loss, although this quantity does not, in reality, indicate the actual loss of silica because of the inorganic material present in all raw cotton. A portion of the water-extractable matter present in the sliver was removed in the treatment, and the impregnation bath increased in solids content with time, up to a limiting equilibrium value.

Samples for the study of silica loss in processing were selected at the beginning of the impregnation treatment and after approximately 10 pounds of sliver had been treated. These were drawn, processed into roving, and spun, after which samples of card sliver, drawing sliver, roving, and yarn were ignited to constant weight in porcelain crucibles. The results of the ash determinations, given in Table II, are the averages of duplicate determinations.

The increase in ash content noted between card sliver and drawing sliver in some samples could not be accounted for but was thought to be due in part to variations in ash content of the original sliver. Although certain discrepancies appear in the data, the greater portion of the results indicate that more silica is lost in the initial drawing operation than in subsequent processes, and also that more is lost as the silica concentration in the treated cotton is increased. These data are in accord with qualitative observations of silica deposition made during the processing operations.

Drafting Difficulties

Two major drafting difficulties were encountered in the processing of Syton-treated sliver. In certain instances the treated samples produced a very irregular drawing sliver, leading to ends down and slubs in the roving, and hard ends appeared in the spinning operation with much higher frequency than is the case in the spinning of untreated cotton. Although the mechanical causes of poor drawing have not been defined, they have been largely eliminated by increasing the back-roll spacing and by the treatment of drawing sliver, which appears to draft more readily than treated card sliver.

Roving produced from Syton-treated cotton at normal twists is much harder and more coherent than comparable roving from untreated cotton. Early in the experiments it was noted that a great many hard ends occurred during spinning of the normally twisted roving, and it was found necessary to decrease the roving twist to produce a softer roving and to minimize drafting difficulties in the spinning operation. Because of the small number of spindles involved in most of the spinning tests, no investigation was made of the effect of roving twist on spinnability. The use of a roving-frame twist gear having ten teeth more than that recommended for use with untreated cotton was found to be quite satisfactory in most tests. This choice was entirely arbitrary, however, and the optimum roving twist is undoubtedly a function of the cotton used and the concentration of silica present.

TABLE III. EFFECT OF PROCESSING TEMPERATURE AND HUMIDITY ON YARN STRENGTH

Sample	Apparent add-on, weight percent of dry cotton	Processing conditions: Drawing	Roving	Spinning	Corrected skein strength* of 11s yarns (lbs.) Twist multiple 2.70	3.15	3.55	3.97
6	2.81	Not redrawn	70°F, 65% R.H.	70°F, 65% R.H.	284	288	286	277
7	3.25	70°F, 65% R.H.	70°F, 65% R.H.	70°F, 65% R.H.	333	323	312	300
8	2.50	First conditioned at 70°F, 65% R.H. Redrawn at 85°F, 37% R.H.	70°F, 65% R.H.	70°F, 65% R.H.	290	293	281	288
9	2.56	85°F, 37% R.H.	85°F, 37% R.H.	85°F, 65% R.H.	302	312	298	277
10	2.66	Not redrawn	85°F, 37% R.H.	85°F, 65% R.H.	287	289	285	280
11	2.62	85°F, 37% R.H.	70°F, 65% R.H.	70°F, 65% R.H.	320	322	315	309
12	2.49	Same as sample 7			306	319	315	308
Control (untreated cotton)		70°F, 65% R.H.	70°F, 65% R.H.	70°F, 65% R.H.	165	227	249	249†

* All breaks made at 65% R.H. and 70°F.
† Interpolated value.

Effect of Spinning-Laboratory Temperature and Humidity on Yarn Strength

It was not possible in all cases to duplicate yarn strengths between identically treated lots of cotton, and for this reason it seemed advisable to examine more closely than had been done previously certain of the processing variables which might influence yarn strength. Two factors were considered: (1) the effect of redrawing treated drawing sliver and (2) the temperature and humidity at which drawing, roving, and spinning were carried out.

A series of identical drawing sliver samples were treated with a Syton dispersion to give as nearly as possible a silica concentration of 2.3 percent, based on the bone-dry weight of the untreated cotton. These samples were processed into 11s yarns under different conditions, as indicated in Table III, and the resultant yarn-skein strengths were determined in the usual way.

The variation in yarn strength in this series of tests appears to be due, in large measure, to differences in add-on. The solution pickup could not be controlled sufficiently closely with the squeeze rolls used to permit the detection of differences in yarn strength which might have been due to variations in the processing conditions. It is felt that the data reported in Table III do not indicate any particular advantage in the processing of Syton-treated cotton at temperatures and humidities other than standard, and that, within the limits of experimental error, the strength values obtained are commensurate with the respective add-on values.

TABLE IV. BLENDING OF TREATED AND UNTREATED COTTON DURING DRAWING

	Number of ends of drawing sliver						
Untreated	6	5	4	3	2	1	0
Treated	0	1	2	3	4	5	6
Calculated silica content, weight percent of dry cotton	0.0	0.39	0.77	1.16	1.54	1.93	2.31
Twist multiple	Corrected skein strength (lbs.)* (10s yarn)						
2.68	154	195	218	233	240	269	276
3.00	190	226	245	256	260	280	302
3.35	218	240	257	*274*	274	*292*	300
3.75	230	252	*269*	264	273	282	*308*
4.10	244	*256*	265	266	268	290	306
4.50	240	255	264	269	*275*	281	283
4.88	*250*	243	252	260	268	271	275
Percent increase in maximum strength over untreated cotton	—	2.4	7.6	9.6	10.0	16.8	23.2

* Figures in italics indicate maximum skein strength.

Blending of Treated and Untreated Slivers at the Draw Frame

To study the possibility of treating only a portion of the cotton with the silica dispersion, and of realizing the same effects previously noted by the incorporation of both treated and untreated cotton in the finished yarn, a study was undertaken of the blending of treated and untreated slivers during the drawing operation.

Drawing sliver impregnated to contain approxi-

TABLE V. BLENDING OF TREATED SLIVERS OF HIGH SILICA CONTENT WITH UNTREATED COTTON

	Sample 13	14	15	Untreated cotton
Silica content of dry treated cotton, weight percent of silica-free cotton	14.80	7.45	2.26	0
Ends of treated sliver	1	2	6	0
Ends of untreated sliver	5	4	0	6
Calculated silica content of blended sliver, weight percent of dry cotton	2.47	2.48	2.26	0
Twist multiple	Corrected skein strength of 11s yarns (lbs.)			
2.57	219	245	279	105
3.02	254	274	307	157
3.48	273	274	311	195
3.99	283	272	299	210
4.53	274	271	286	222
5.09	267	265	276	211

TABLE VI. EFFECT OF pH OF BATH ON STRENGTH OF SYTON-TREATED YARNS

Initial bath composition, weight percent	pH of bath 3	4	5	6	7	8	9
Impregnation data							
SiO_2	1.25	1.25	1.25	1.25	1.25	1.25	1.25
Aerosol OT	0.50	0.50	0.50	0.50	0.50	0.50	0.50
NH_3	0.08	0.08	0.08	0.08	0.13	0.15	0.15
H_3PO_4	0.23	0.28	0.26	0.25	0.23	0.22	0.19
Total solids	2.22	2.32	2.19	2.26	2.21	2.19	2.13
Corrected pick-up, percent of dry cotton	66	75	85	76	70	68	72
Apparent add-on, percent of dry cotton	1.48	1.74	1.86	1.71	1.55	1.50	1.53
Spinning data							
Twist multiple	Corrected skein strength for 11s yarn (lbs.)						
2.68	262	267	266	254	248	245	258
3.13	271	283	286	278	285	262	279
3.57	270	281	290	279	275	275	283
4.02	262	268	272	267	260	260	270

TABLE VII. SKEIN IMPREGNATION

Solution composition	Weight percent
Syton W-20	12.90
Aerosol OT	0.25
Bath solids	2.37
Solution pickup, percent of dry cotton	65.7
Apparent add-on, percent of dry cotton	1.50

Remarks	Skein strength (lbs.)
Untreated cotton	160
Water-immersion control	161
Syton impregnation	197
Yarn from treated sliver at 3.50 twist multiple	252

TABLE VIII. COMPARISON OF YARN AND SLIVER TREATMENTS WITH SYTON W-20

Nominal-twist multiple	Impregnation Method*	Time (min.)	Apparent add-on, weight percent of dry cotton	Corrected skein strength for 10s yarns (lbs.)
3.00	Untreated	0	0.00	192
	Skein A	10	1.82	227
		30	1.88	199
		60	1.86	216
		1,000	2.01	220
	Skein B	30	1.77	242
	Skein C	30	1.86	201
	Sliver-treated	*	1.78	335
3.50	Untreated	0	0.00	226
	Skein A	10	1.82	237
		30	1.83	234
		60	1.90	241
		1,000	1.91	234
	Skein B	30	1.74	244
	Skein C	30	1.81	233
	Sliver-treated	*	1.78	318
4.00	Untreated	0	0.00	233
	Skein A	10	1.83	243
		30	1.85	247
		60	1.81	247
		1,000	1.84	234
	Skein B	30	1.80	243
	Skein C	30	1.80	228
	Sliver-treated	*	1.78	295

* Skein method A: Immersion at room temperature and atmospheric pressure. Skein method B: Immersion at 81°C and atmospheric pressure. Skein method C: Immersion in bomb at room temperature and 1,500 p.s.i. under nitrogen atmosphere for 30 minutes. Sliver-treated: The impregnation time in the sliver treating apparatus was approximately 0.02 to 0.03 minutes.

mately 2.3 percent silica was blended with untreated drawing sliver at the draw frame in successive tests in which the number of treated ends was varied from zero to 6. The blending and yarn-strength data are given in Table IV and it appears that the Syton-treated cotton in the blend contributes to increased yarn strength but not in direct proportion to the silica content of the blend. The greater percentage increases in cases where four, five, and six treated ends were used indicate that uniformity of the treatment may have had some influence in the strength increase realized.

To confirm the indication that uniformity of

treatment may affect yarn-strength increases, an additional experiment was undertaken in which drawing sliver containing silica in relatively high concentration was blended with untreated sliver as before. Untreated cotton and cotton impregnated to contain approximately 2 percent silica were used as controls in this test. Samples 13 and 14, Table V, were treated with undiluted Syton W-20 and with a mixture of 50 percent Syton and 50 percent water, respectively. Sample 13 was then blended as a single end with five ends of untreated drawing sliver at the draw frame, and sample 14 was treated similarly, using two ends of the treated cotton and four of the untreated material to give approximate silica concentrations of 2.5 percent in both blended slivers. The results of the spinning tests on the blended slivers are shown in Table V, together with spinning data for a uniformly treated sliver containing slightly less silica (sample 15) and for untreated cotton.

It is apparent from the yarn-strength data that the uniformity of treatment is an important factor in the development of maximum yarn strength. Although both blended slivers contained more silica than did sample 15, the maximum yarn strength of the latter was significantly greater than that obtained in spinning either of them.

Effect of Bath Acidity on Yarn Strength

To determine whether the pH of the impregnation bath influenced the strength of yarns from Syton-treated cotton, a series of treatments was carried out in which the pH of the dispersion was varied between the limits of 3 and 9.

To each bath was added sufficient ammonia and phosphoric acid to adjust the initial pH to the desired value. As much additional phosphoric acid or ammonia in concentrated solution was added from a burette during the impregnation as was required to maintain a constant solution pH, as measured continuously with a Beckman Model G pH meter.

Impregnation and spinning data are presented in Table VI. Insufficiently closely controlled pickup makes interpretation of the results difficult. Although an apparent maximum in yarn strength was attained with a bath having a pH of 5, this strength increment was probably due to the higher silica add-on. On the whole it would appear that the effects of Syton impregnations are not greatly influenced by pH variations in the range studied.

Yarn Treatments

In order to evaluate the effect of Syton impregnations of yarn in skein form, samples of untreated 11s yarn were spun at a twist multiple of 3.00, reeled into 120-yard skeins, and treated by immersion in Syton W-20 solution for a period of 1 hour at room temperature. Excess solution was removed by passing the skeins between squeeze rolls. Solution pickup was determined by the weighing of individual skeins before and after treatment. The skeins were then allowed to air-dry and were broken on the Scott J-2 Tester according to ASTM Designation D180-42. An identical set of skeins was subjected to the same immersion period in a 0.25-percent Aerosol OT solution in water as a control. The skein impregnation and test data are given in Table VII. Skein strengths are the average corrected results of ten breaks.

The preliminary tests indicated that skein impregnation with Syton does not produce so great a yarn-strength increase as has been realized in sliver impregnation. Although the strengths of yarn-treated and sliver-treated samples are not strictly comparable since the yarn-treated sample was spun at a twist multiple of 3.00 and the sliver-treated sample was spun at a twist multiple of 3.50, these twists are sufficiently close that it may be safely assumed that the strengths would not have been identical even if they had been the same.

It was thought possible that the cause of the discrepancy between the yarn strengths may have been incomplete penetration of the yarn by the treating solution. In order to determine whether this was the case, further skein impregnations were conducted in which the duration of immersion was varied. Tens yarns spun at nominal twist multiples of 3.0, 3.5, and 4.0 were reeled into standard 120-yard skeins, which were then soaked in a dispersion of Syton W-20 containing 2 percent silica, squeezed, and allowed to air-dry. The period of immersion was varied from 10 to 1,000 minutes. In addition to these impregnations carried out at room temperature and atmospheric pressure, similar experiments were performed at 81°C at atmospheric pressure and (in a bomb) at room temperature under 1,500 pounds pressure per square inch under nitrogen for 30 minutes. Ten skeins were treated in each case.

Contrary to expectations, the pickup of solution (and hence the add-on of silica) was not increased by longer immersion, by immersion at the higher

temperature, or by immersion under high pressure. Neither did the less tightly twisted yarns pick up more of the solution than those spun at conventional twists.

Results of these experiments are presented in Table VIII. Data for untreated and sliver-impregnated cotton are included for comparison.

It is apparent that the tensile strengths of the impregnated skeins were in no case as high as those obtained from sliver-impregnated cotton. The best results of the skein impregnations were obtained at 81°C and there is apparently no advantage in increasing the impregnation time over 10 minutes when treatment is carried out at room temperature. The increase of pressure to 1,500 pounds per square inch was also without effect on the final yarn strength.

The results of the yarn treatments lead to the conclusion that yarn impregnation does not offer the same possibilities for yarn-strength increase as does impregnation prior to the completion of the spinning operation.

Summary

The work described in this paper covers further studies in the treatment of cotton in sliver form with colloidal dispersions of Syton.

The treatment of drawing sliver appears to offer some advantages over the treatment of card sliver, in so far as ease of subsequent drafting operations and final yarn strength are concerned. Shedding of silica from the treated sliver during drafting was investigated, and it was found that the greatest portion of the silica loss occurred during drawing. Silica loss was also found to increase with increasing silica concentration in the treated sliver.

It has been observed that the drafting of treated slivers is somewhat more difficult than the drafting of untreated ones, and certain modifications in drawing-roll spacings and roving twists have been found to facilitate drafting of silica-treated cotton. The variation of spinning-laboratory temperature and humidity during the processing of treated cotton was found to affect spinning performance and final yarn strength to an insignificant degree.

Studies have been made of the blending of treated and untreated slivers during drawing, and the results of these tests indicate that, at comparable silica concentrations, higher-strength yarns are produced when the silica is uniformly distributed in the cotton during treatment. Although the treatment of yarns produces some increase in strength, it is of a much lower order of magnitude than that encountered in the sliver impregnation process even when the yarns treated are of low twist.

Conclusions

From the results of the investigations reported, the following conclusions have been drawn:

1. The treatment of drawing sliver may be accomplished in a continuous single-strand operation, and treated drawing sliver drafts with less difficulty than comparably treated card sliver. Yarn-strength increase is somewhat greater for treated drawing sliver than for treated card sliver.

2. Some of the silica in treated sliver is removed during drawing, roving, and spinning operations. The greatest amount is lost during drawing.

3. It is advisable to produce roving of treated cotton with minimum twist to facilitate drafting on the spinning frame and minimize hard ends.

4. Variations in pH of the treating bath and temperature and humidity in the spinning laboratory were without effect on final yarn strength over the ranges investigated.

5. Blending of treated and untreated slivers during drawing does not produce so great a yarn-strength increase as that realized in the uniform treatment of slivers.

6. Although the treatment of yarn with Syton dispersions produces some strength increase, it is of a much lower order of magnitude than that encountered in the sliver treating process.

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Location of Melamine Resin in Treated Wool Fibers

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Abstract

A dye-staining and sectioning technique has been developed which permits, for the first time, a direct, visual determination of the location of melamine resin in treated wool fibers. The resin appears to be distributed uniformly within the fiber structure. Techniques are described and the results are illustrated by photomicrographs (one in color).

EVER since the first resin treatments were successful in modifying the properties of textile materials, the problem of determining where the resin is deposited has been an obstacle to the fullest theoretical and practical considerations of resin treatment.

Previous studies on "zoning" in the resin treatment of textile materials have yielded satisfactory methods for the detection of urea and melamine resins when cellulosic materials were considered [5, 6, 9]. Because of the close structural similarity between wool and the melamine resins, it has been impossible, heretofore, to determine with certainty the location of melamine resins applied to wool [2].

It is known that acid dyes will stain both wool and melamine resins, and that the rate at which wool can be dyed is changed after resin treatment [7]. This fact, presumably, has been the basis for some attempts to locate the melamine by studying the rate of dyeing of treated and untreated wool. The difficulty of locating the resin by using dyes has been mentioned [2, 5, 7]. Allusions to the uniform distribution of melamine resin within wool fibers have been made, but direct evidence for this has been lacking [4, 5].

Investigations in the Monsanto laboratories have provided for the first time a direct, visual method for determining the location of melamine resin in treated wool fibers. It has been found that the use of Aqua Plastic Red with the technique described below will differentiate between wool and melamine.

Experimental Data

For all tests, standard wool fabric supplied by the American Association of Textile Chemists and Colorists was used. The melamine resin, Resloom HP, was applied to the wool fabric from water solution in the conventional manner. Aqua Plastic Red, as a 10-percent solution in water, was the staining medium.

Small swatches of melamine-treated and untreated wool fabric were placed in 300 ml. of dye solution contained in a 500-ml. flask connected to a reflux

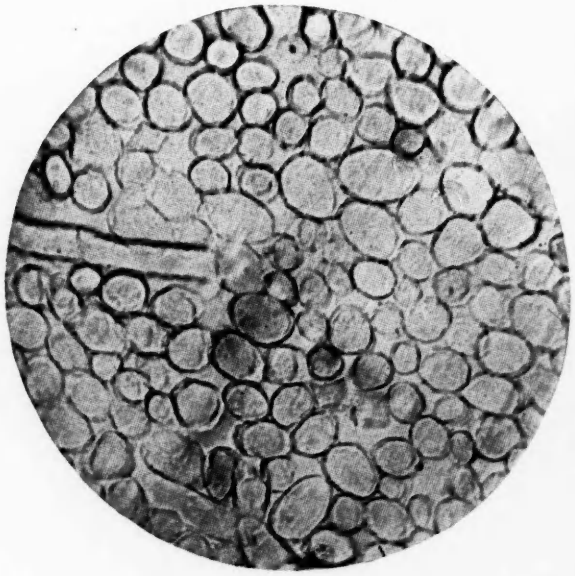

FIG. 1. *Untreated wool fibers after dyeing and washing. (This photomicrograph is reproduced in black and white because the amount of stain within the fiber is so slight as to be hardly discernible in the colored transparency and would not be reproducible in a color print. The darker areas in this illustration indicate portions in which a slight stain appears. However, in photographing in black and white unavoidable overemphasis is given to the amount of stain present.*—EDITOR)

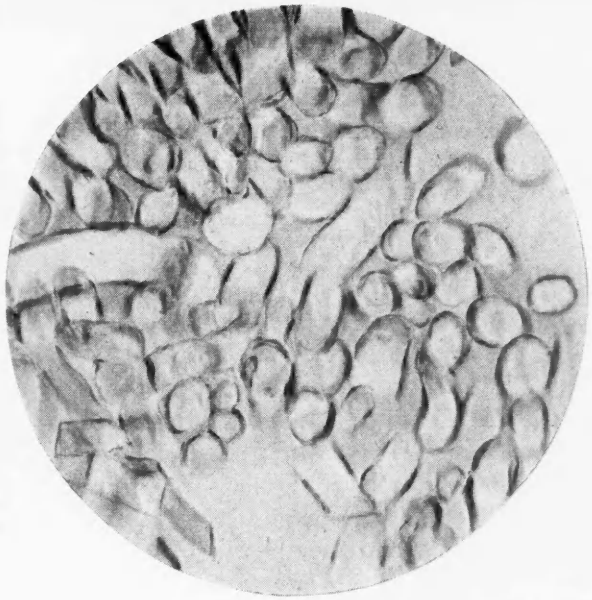

FIG. 2. *Melamine-treated wool fibers after dyeing and washing.*

condenser. The flask and its contents were then heated on a boiling-water steam bath for 1 hour after refluxing began in the flask. At the end of this period, the flask was cooled in an ice bath and the swatches were removed directly into about 200 ml. of cold distilled water, and given three cold rinses with stirring to remove surface dye.

The swatches were then washed free of excess dye by being placed in successive portions of simmering distilled water until, in a wash, practically no dye was removed after about 10 minutes of simmering. Swatches were then dried in a circulating-air oven at 60°C.

Suitable, small pieces of each swatch were cut parallel to the threads. Each small piece was then mounted in the Schwarz fiber microtome and sections 20 microns thick were cut. This thickness was chosen to provide sections thin enough to transmit light readily but thick enough to show color well and to be cut and handled easily. The use of this type of microtome has been described adequately [1, 3, 8]. Sections were mounted on slides in Canada balsam diluted with xylene to the consistency of light syrup. The various sections were examined at a magnification of 200 × under a microscope fitted with color-corrected lenses. Photomicrographs were taken on 5-by-7-inch Type B Kodachrome film.

It was found advisable to prepare sections of fabric rather than of bundles of fibers. In this way cross, oblique, and longitudinal sections of fibers could be observed together. Thus, the effect of melamine treatment could be studied more completely than is possible with the conventional cross sections.

Results

Under the conditions used, the dye stains melamine resin but not the untreated wool fibers. Thus, when color is present inside the fibers as viewed under the microscope, this color serves to indicate the presence of melamine resin inside the fibers. Figure 1 shows a section of untreated wool after dyeing and washing. Figure 2 shows a section of melamine-treated wool after dyeing and washing.

It should be pointed out that this method of detection is qualitative and should not be used to judge, other than roughly, the amount of resin present.

Under the microscope, direct observations of the various sections led to the following conclusions concerning melamine resin applied to wool fabric from aqueous solution:

1. It is possible to use a dye-staining method for determining the location of melamine resin applied to wool fibers.

2. The melamine resin appears to be distributed uniformly within the wool fiber structure.

3. There is no obvious change in scale structure of the wool fibers after treatment with melamine resin.

Acknowledgment

The cooperation of Dr. D. H. Powers and the Textile Chemical Department of Monsanto Chemical Company in this work is deeply appreciated.

The Aqua Plastic Red was supplied by the Great American Color Co.

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Introduction of Amino Groups into Cotton Fabric by Use of 2-Aminoethylsulfuric Acid

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SEVERAL attempts to introduce amino groups into cellulose or into cotton fabrics in a practical manner are recorded in the literature. The attempt most pertinent to the present investigation is that of Hartmann [2], who reacted soda cellulose or cellulose treated with 18-percent sodium hydroxide with 2-chloroethylamine and similar compounds. Hartmann's procedures are long, requiring from 1 to 12 hours, and some of them require the use of organic solvents. When the author tried to put amino groups into cotton fabric by means of 2-chloroethylamine hydrochloride, no appreciable amount was introduced either by treating the fabric with 20-percent sodium hydroxide followed by the compound or by wetting the fabric with an aqueous solution of the compound, drying, and then treating with 20-percent sodium hydroxide. This was also true of 2-bromoethylamine hydrobromide. However, enough amino groups to give a good dyeing with an acid wool dye could be introduced if the fabric was treated with an aqueous solution of 2-chloroethylamine hydrochloride, dried, and then put into 50-percent sodium hydroxide. It was necessary for the fabric to remain in the sodium hydroxide for 15 minutes in the cold, but if the 50-percent sodium hydroxide was held at about 100°C, only 1 minute was required. Subsequent experimentation showed that this method, although quick and convenient, introduced only about 0.2 percent of nitrogen into the fabric.

Since there seemed to be some possibility that 2-aminoethylsulfuric acid might be effective, this relatively inexpensive compound was tried. When used like the chloro compound it was slow-reacting, requiring more than 15 minutes in the hot 50-percent sodium hydroxide to give a fabric showing dyeing with an acid wool dye. When, however, this compound was put into mercerization-strength sodium hydroxide solution (17- to 25-percent), and the cotton was moistened with this solution, dried at about 100°C, held at this temperature for a while, washed to remove the sodium hydroxide, and dried, the fabric dyed dark with an acid wool dye. Since 2-aminoethylsulfuric acid ($NH_2 \cdot CH_2 \cdot CH_2O \cdot SO_2 \cdot OH$) may be readily prepared by the action of fuming sulfuric acid on ethanolamine, and since the process seemed simple and effective, it was studied in some detail.

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Materials

The fabric used in most of the experiments was a commercial broadcloth, 4 yards per pound, 108-by-60 thread count, that had been singed, desized, kier-boiled, and bleached. The 2-chloroethylamine hydrochloride was made by the procedure of Ward [4]. The 2-aminoethylsulfuric acid was made by the dropwise addition of 20 ml. of fuming sulfuric acid, 20 percent excess SO_3, to 10-ml. portions of ethanolamine, cooled in ice-salt mixture. After addition of a little water to this reaction mixture, the substance was precipitated with an excess of ethyl alcohol, separated by filtration, redissolved in water, and reprecipitated with alcohol. The yield was about 15 g. This method of preparation is essentially that of Fränkel and Cornelius [1].

Experiments with 2-Chloroethylamine

In one series of experiments 8-percent and 2.4-percent aqueous solutions of 2-chloroethylamine hydrochloride were applied to 12-by-30-cm. areas of fabric stretched on stainless-steel beakers. Uptake averaged 6.5 g. of the solution. After drying at 105°C, the beaker and fabric were pushed into a bath of saturated (50-percent) sodium hydroxide held at 100°C. After 1-, 3-, and 10-minute intervals the beaker and fabric were taken from the bath,

TABLE I. AMOUNT OF NITROGEN (IN PERCENT OF DRY WEIGHT) INTRODUCED INTO COTTON FABRIC BY TREATMENT WITH 2-AMINOETHYLSULFURIC ACID UNDER VARIOUS CONDITIONS

Solution	Percentage of: NaOH	Percentage of: 2-aminoethyl-sulfuric acid	Temperatures and times of heating: 90°C, 20 min. % N	90°C, 40 min. % N	100°C, 20 min. % N	100°C, 40 min. % N	110°C, 20 min. % N	110°C, 40 min. % N	70°C, 9 hr. % N
A	25	10	0.15	0.37	0.23	0.56	0.47	0.62	0.59
B	25	3	0.03	0.20	0.13	0.23	0.20	0.24	0.25
C	50	10	0.07	0.31	0.13	0.30	0.34	0.36	0.28
D	50	3	0.08	0.20	0.16	0.18	0.20	0.21	0.15
Control	25	0	0.01					0.02	0.02
Control	50	0	0.01					0.02	0.02

washed thoroughly in running tap water and in distilled water, and dried. In some cases the dried fabric was removed from the beaker just before it was put into the hot sodium hydroxide solution. The total nitrogen values on the dry basis were 0.23 ± 0.02 percent for the fabrics treated with the 8-percent solution and 0.10 ± 0.02 for those treated with the 2.4-percent solution. Control fabrics treated with water, dried, and dipped into the hot sodium hydroxide solution averaged 0.02 ± 0.02 percent. These values were independent of the time in the hot sodium hydroxide. Removal of the fabric from the can just prior to putting it into the hot sodium hydroxide solution did not alter these values. Dyeing with Kiton fast red was in agreement with the total nitrogen values. Breaking strength of the fabrics averaged 50 lb. for the 1-minute hot sodium hydroxide treatment, 49 lb. for the 3-minute treatment, and 47 lb. for the 10-minute treatment. Untreated control fabrics averaged 51 lb. All values are corrected to the basis of 100 warp threads.

Similar experiments with 2-bromoethylamine hydrobromide showed this compound to be slightly less effective than the chloro compound on an equimolecular basis.

Experiments with 2-Aminoethylsulfuric Acid

The ranges of concentrations, durations of treatment, and temperatures chosen were selected on the basis of preliminary exploratory experiments. These experiments indicated that a favorable concentration of sodium hydroxide was 25-percent or perhaps 50-percent. A satisfactory concentration of the 2-aminoethylsulfuric acid was about 10-percent. Good dyeings with an acid wool dye were obtained, however, with 3-percent of the 2-aminoethylsulfuric acid. A favorable drying and heating temperature was about 100°C. It was decided to investigate these conditions of treatment in greater detail.

The fabric was cut into 12-cm. strips lengthwise the warp. These strips were pinned into sleeves 32 cm. in circumference and slipped over stainless-steel beakers of 32-cm. outside circumference. A 12-by-30-cm. area (3.7 g. dry weight) of the fabrics was then moistened with the solutions by means of glass-wool swabs and the beaker and fabric were placed in a forced-draft oven at various temperatures for various times. After baking, the fabrics were washed at least 45 minutes on the beaker in running tap water and then with two or more changes of distilled water.

The solutions used were: (*A*) 18.75 g. of sodium hydroxide, 48.75 ml. of water, and 7.5 g. of 2-aminoethylsulfuric acid; (*B*) 18.75 g. of sodium hydroxide, 54.0 ml. of water, and 2.25 g. of 2-aminoethylsulfuric acid; (*C*) 67.5 g. of 50-percent sodium hydroxide and 7.5 g. of 2-aminoethylsulfuric acid; (*D*) 2.25 g. of 2-aminoethylsulfuric acid and 72.75 g. of 50-percent sodium hydroxide. Appropriate controls were included. It was necessary to apply solutions *C* and *D* hot in order to keep the material in solution. The take-ups of solutions *A* and *B* were about 8.5 g., and for *C* and *D* about 12.5 g.

The introduction of amino groups was measured by Kjeldahl nitrogen determinations. For this, the material was digested in the usual manner using mercury as a catalyst. The ammonia was distilled into an excess of dilute acid and determined colorimetrically using Nessler's reagent. Moisture determinations were made so that the nitrogen values could be converted to the dry basis. Breaking strengths were determined on three strips of each fabric and

TABLE II. BREAKING STRENGTH (IN POUNDS PER 100 WARP THREADS) OF COTTON FABRIC TREATED WITH 2-AMINOETHYLSULFURIC ACID UNDER VARIOUS CONDITIONS *

Solution	Percentage of: NaOH	Percentage of: 2-aminoethylsulfuric acid	Temperatures and times of heating: 90°C, 20 min. Lb.	90°C, 40 min. Lb.	100°C, 20 min. Lb.	100°C, 40 min. Lb.	110°C, 20 min. Lb.	110°C, 40 min. Lb.	70°C, 9 hr. Lb.
A	25	10	55	45	45	49	51	48	51
B	25	3	48	49	48	50	48	47	51
C	50	10	44	48	49	45	46	44	48
D	50	3	44	44	42	41	46	40	47
Control	25	0	48					46	54
Control	50	0	50					40	46

* The untreated fabric averaged 51 pounds per 100 warp threads.

reduced to the basis of 100 warp threads. The results are summarized in Tables I and II. Dyeing tests with Kiton fast red were in agreement with the total nitrogen values. The fabrics that contained 0.5 to 0.6 percent nitrogen dyed a dark red and those with 0.2 percent nitrogen dyed a lighter red.

Taking into consideration both total nitrogen values and the breaking-strength values, the condition of choice appears to be 25-percent sodium hydroxide with heating for 40 minutes at 100°C. Breaking-strength values (15 breaks) on the fabric treated in this way averaged 48 lb. per 100 warp threads. The untreated fabric averaged 51 lb. (15 breaks). The treated fabric had a cuprammonium fluidity of 9 rhes, whereas the fluidity of the untreated fabric was 3.5 rhes. Tire cord aminized with 2-aminoethylsulfuric acid showed no significant change in breaking strength.

For the purpose of testing the effect of the concentration of sodium hydroxide further, solutions containing 10-percent 2-aminoethylsulfuric acid in 30-, 25-, 23-, 21-, 19-, and 17-percent sodium hydroxide were tried. The total nitrogen values obtained on the dry basis were 0.51, 0.59, 0.57, 0.54, 0.47, and 0.46 percent, respectively. This shows that 25-percent sodium hydroxide is the concentration of choice, although small variations from this concentration are of little importance.

Increasing the heating time beyond 40 minutes does not greatly increase the percent of nitrogen, since heating 16 hours at 100°C using 10-percent 2-aminoethylsulfuric acid in 25-percent sodium hydroxide gave only 0.64 percent of nitrogen as compared with 0.56 percent for the 40-minute period. The fabric heated for 16 hours had a breaking strength of only 37 lb. per 100 warp threads. It will be noted from Table I that the heating temperature may be lowered to 70°C if the time is extended to 9 hours. However, in other experiments heating at 50°C for 45 hours gave a nitrogen value of only 0.24 percent and heating at 40°C for 71 hours gave 0.18 percent of nitrogen. Allowing the wetted fabric to stand in a closed container for several hours before baking did not increase the nitrogen value.

The 2-aminoethylsulfuric acid need not be isolated. In one experiment 10 ml. of ethanolamine were treated with 20 ml. of fuming sulfuric acid in the usual manner. The reaction mixture was diluted with water and neutralized with strong sodium hydroxide solution. It heated and reacted vigorously. After cooling, 58 g. of solid sodium hydroxide were added, followed by water to make a total of 230 g. Although much sodium sulfate separated from this solution, the supernatant liquid was quite effective. A fabric was obtained which gave a dark dyeing reaction after treatment with this solution. Its total nitrogen content on the dry basis was 0.52 percent. After the solution had stood for 3 weeks at room temperature, a fabric containing 0.41 percent nitrogen was obtained by a similar treatment.

The treatment with 2-aminoethylsulfuric acid and sodium hydroxide works on viscose rayon. Acetate rayon must be soaked in the solution for about 40 minutes to deacetylate it prior to baking.

Repeated treatments of the same piece of cotton fabric with 2-aminoethylsulfuric acid have been tried. Parchment-like material having some fabric structure was obtained. Its nitrogen content was 1.84 percent on the dry basis.

Sodium hydroxide solutions containing 2-aminoethylsulfuric acid may be used to print patterns on fabrics. If used in connection with another sodium

hydroxide solution containing monochloroacetic acid [3], acid carboxyl groups may be introduced into some portions of the fabric while amino groups are introduced into other portions. When fabrics so printed are dyed with a basic dye like methylene blue and then with an acid dye like Kiton fast red, two-colored printing results.

Amino groups can also be introduced into raw cotton fiber by treatment with 2-aminoethylsulfuric acid. Raw cotton fiber, 65 g., in the form of picker lap, was moistened thoroughly with a solution composed of 35 g. of sodium hydroxide, 88 ml. of water, 14 g. of 2-aminoethylsulfuric acid, and 3 ml. of a commercial wetting agent of the cresylic-acid type. The cotton was then spread evenly on trays of a forced-draft drying oven and heated at 100°C for 1 hour and 10 minutes. The material was washed thoroughly with water, soured with dilute acetic acid, washed again, and dried. It contained 0.75 percent of nitrogen on the dry basis. It was spun into yarn, which was used as filling in a fabric having untreated cotton for warp. When this fabric was dyed with a chrome wool dye, the aminized filling dyed red, while the warp threads of the ordinary cotton did not take the dye but remained white.

In another experiment with raw cotton in the form of picker lap, 10 lb. of cotton were treated in batches weighing about 1.5 lb. each. The solution used was composed of 6,300 ml. of water, 2,500 g. of sodium hydroxide, 1,000 g. of 2-aminoethylsulfuric acid, and 200 ml. of the commercial wetting agent. After wetting in the solution, each batch was centrifuged at 3,000 r.p.m. in the basket head of a large centrifuge. The effluent of each batch served to wet the subsequent batch. The weight of the cotton after centrifuging was 25 lb. The moist cotton was spread on trays and heated in drying ovens for 3 hours at 90° to 110°C. The cotton was washed free of sodium hydroxide in the basket head of the centrifuge and then washed with a 0.5-percent emulsion of peanut oil, which acted as a softener. After centrifuging, the washed cotton was oven-dried. It gave a dark dyeing with a wool dye.

Amino groups can be introduced into cotton in the form of yarn by treatment with 2-aminoethylsulfuric acid. A sample of yarn was wound on a stainless-steel beaker and moistened with a solution composed of 5 g. of sodium hydroxide, 13 ml. of water, 2 g. of 2-aminoethylsulfuric acid, and 0.4 ml. of the commercial wetting agent. It was heated in a drying oven at 100°C for 40 minutes. After washing and drying it contained 0.73 percent of nitrogen on the dry basis. Some of the yarn was sewed on pieces of wool fabric and dyed with a chrome wool dye. By controlling the acidity of the dye bath with formic acid, a match in shade between the wool fabric and the aminized yarn could be obtained.

Properties of Fabric Treated with 2-Aminoethylsulfuric Acid

The outstanding property of the fabric treated with 2-aminoethylsulfuric acid and sodium hydroxide is its dyeing with acid dyes, a property not possessed by ordinary cotton fabric. The aminized fabric also dyes well with a chrome dye such as alizarin blue black B (Index No. 1085), whereas the untreated fabric does not. The treated fabric also shows increased affinity for direct cotton dyes.

The amino groups of the treated fabric exhibit the expected properties of amino groups, the outstanding one being the capacity to take up acids. The aminized fabric takes up hydrochloric acid from dilute solution in amounts commensurate with its amino content measured by total nitrogen values. It is dyed yellow with picric acid. When placed in dilute phosphoric acid it takes up, and retains after thorough washing, enough of this acid to give a black ash which shows no afterglow when the fabric is burned. When the fabric is placed in 1-percent sodium dichromate solution and thoroughly washed it retains enough chromic acid to become a brownish yellow; untreated fabric, or fabric treated with sodium hydroxide alone, remains white. The aminized fabric treated with sodium dichromate showed rot-resistance in a soil-burial test. Rot-resistance may also be obtained by treating the aminized fabric with a 1-percent solution of mercuric chloride followed by washing with water. Aminized fabric so treated was still strong after burial for 7 weeks, while ordinary cotton fabric treated with mercuric chloride in the same way rotted in 2 weeks. Fabric with no treatment rotted in 1 week. When placed in 0.5*N* potassium permanganate, acidified with a little acetic acid, the aminized fabric turns a dark brown. This color is not removed by thorough washing. These are but a few examples of the possibilities of the fabric. The attached amino

groups will no doubt serve as an effective means for the attachment of other substances.

Discussion

It should be emphasized that 2-aminoethylsulfuric acid is not just an equivalent for 2-chloroethylamine and similar halogenated amino compounds. They cannot be used interchangeably. Aside from its greater effectiveness and economy, 2-aminoethylsulfuric acid has certain properties not possessed by 2-chloroethylamine, the more important of which are its nonvolatility and its acid nature, which hold it firmly in the sodium hydroxide solution. It dissolves readily in sodium hydroxide solution and does not separate as does 2-chloroethylamine in that solution. It is also quite stable in sodium hydroxide solutions at room temperature.

The amino groups are presumably introduced into the fabric as amino ethoxyl groups, replacing some of the hydroxyl groups of the cellulose. This involves an ether linkage which should be quite stable to hydrolytic agents. It may be calculated that 0.56 percent of nitrogen on the dry weight basis is equivalent to about one amino group for every 15 glucose units in the cellulose molecule. The efficiency of the introduction of amino groups based on the take-up of the fabric is about 25 percent for 2-aminoethylsulfuric acid, as compared with 13 percent for 2-chloroethylamine hydrochloride.

Summary

1. Amino groups may be introduced into cotton fabric by use of 2-aminoethylsulfuric acid. The fabric is moistened with a 10-percent solution of this substance containing 25-percent sodium hydroxide. It is then heated for 40 minutes at 100°C in a drying oven and washed thoroughly. The nitrogen content of fabric so treated is about 0.56 percent of the dry weight. It dyes readily with acid wool dyes. Other properties of this aminized fabric are described.

2. Amino groups can also be introduced into cotton fabric by use of 2-chloroethylamine, but a different method must be used from that employed for 2-aminoethylsulfuric acid. This method consists of a treatment of the fabric with an aqueous solution of 2-chloroethylamine hydrochloride, drying, placing the dried fabric into hot 50-percent sodium hydroxide solution for 1 minute or more, and washing out the sodium hydroxide with water. The 2-chloroethylamine is more expensive and appears to be less efficient than 2-aminoethylsulfuric acid.

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Laboratory Techniques

This department in TEXTILE RESEARCH JOURNAL *is a special section for notes on laboratory techniques peculiar to textile testing and research which would ordinarily not be of sufficient importance or length for publication as a formal* JOURNAL *paper.*

It is intended to encourage free exchange of helpful modifications of existing tests or apparatus for special purposes and to bring to light newly developed methods and devices. The time spent in standardizing present tests and in developing new ones for the multitude of problems facing the textile technologist would be considerably reduced if information of this sort were made available. Therefore the JOURNAL *invites contributions from its readers.*

In accordance with the established practice, all papers will be submitted to competent judges before being accepted for publication.

Simplified Method for Evaluating the Dimensional Stability of Tubular Knit Fabrics

Louis I. Weiner*

CURRENT shrinkage measurement specifications require that after extraction laundered specimens be dried flat in an oven, following which they are sprayed and pressed before remeasuring.

In connection with these procedures, two major causes of excessive variation have been noted: (1) inability to dry without distorting the specimen, and (2) the dependence of results on individual manipulative techniques in measurement. An estimate of the variation of the currently accepted measuring method † is given in Table I. Measurements were made of the shrinkage of a medium-weight ribbed underwear tubing, knit from a single 20s combed cotton yarn, weighing approximately 6.5 ounces per square yard. A standard cotton-laundering procedure was used.†

Two possible means of reducing this great variation are suggested: (1) by effecting perfectly tensionless drying through use of a rotary drier, and (2) by measuring the fabrics after application of constant light tension across the width of the fabric to eliminate wrinkles.

Experiments were made on three typical rib-knit constructions representing a range of weights and yarn sizes within which most underwear fabrics would fall. A summary of the characteristics of the three constructions is given in Table II.

Specimens from each sample were randomized with respect to position along the length of the respective bolts and then were divided into two groups. One group was measured flat ‡ in the conventional manner and the other was measured using a tension (bar) device. Both groups were laundered and then dried in a rotary drier for 30 minutes at 130°F. The specimens were then allowed to attain moisture equilibrium under standard conditions of temperature and R.H. Measurements before and after laundering were made by three individuals to obtain an indication of personal variations.

The increased precision effected by the substitution of rotary-drying for oven-drying was clearly significant. For example, the standard deviations listed in Table I, which were 1.8 percent for the wales and 2.8 percent for the courses when conventional oven-drying was employed, were reduced to 0.6 percent for the wales and 0.4 percent for the courses (in samples of the same weight) after rotary-drying (Table III).

Measurements after the application of tension were accomplished by means of the bar device illustrated

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† "Textiles: General Specifications, Test Methods," CCC–T–191a (Section XIV), 1945.

‡ Accomplished by measuring the actual width of the specimens at four positions and the distance between three sets of 18-inch markers in the length. All measurements were made after the specimens had attained moisture equilibrium in an atmosphere of 65% R.H. at 70°F.

TABLE I. SHRINKAGE OF MEDIUM-WEIGHT KNIT FABRIC MEASURED AFTER OVEN-DRYING

Average shrinkage (%)		Standard deviation (%)	
Wales	Courses	Wales	Courses
12.7	−4.3	1.8	2.8

NOTE: Averages are based upon 10 specimens; minus sign indicates elongation.

TABLE III. SHRINKAGE OF KNIT FABRICS MEASURED AFTER ROTARY-DRYING

Fabric	Average shrinkage (%)		Standard deviation (%)	
	Wales	Courses	Wales	Courses
Lightweight	18.3	0.4	0.9	0.6
Medium-weight	16.0	−1.0	0.6	0.4
Heavyweight	15.2	−6.4	0.8	1.0

in Figure 1. With the tubing suspended from a bar supported by the uprights, a second bar was inserted into the tubing and lowered gradually to the bottom, where it provided an even tension across the width of the material. The length of the suspended tubing was then determined by measuring the distance between 18-inch markers (three pairs) while the width dimensions were taken at four places, measured from the top of the upper bar to the bottom of the lower bar. After initial measuring, the specimens were laundered with no effort being made to open or spread the material before it was placed in the washer, so as to avoid excessive distortion.

With rotary-drying clearly established as a necessary feature of the test method, the F test was used to compare the variability of the two measuring techniques and the coefficients of variation were used to compute the average sample size required for a precision in measurement of 95 percent and an accuracy of approximately 5 percent.

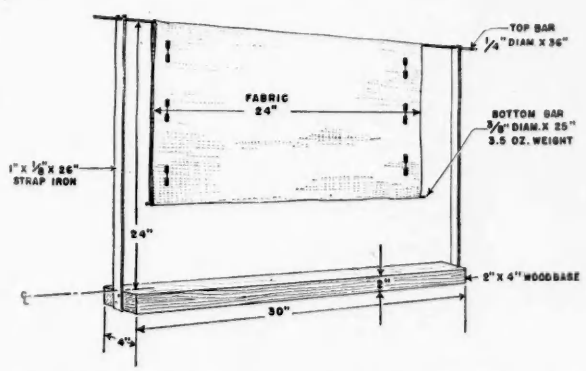

FIG. 1. *Bar device for measuring the shrinkage of knit tubing.*

TABLE II. PHYSICAL CHARACTERISTICS OF KNIT GOODS

Fabric	Weight (oz./sq. yd.)	Yarn no.	Wales	Courses	Knit	Yarn
Lightweight	5.2	1/30s	30	34	Rib 1×1	Combed
Medium-weight	6.5	1/20s	24	30	Rib 1×1	Combed
Heavyweight	10.6	1/10s	20	24	Rib 1×1	Combed

TABLE IV. VARIANCES SHOWING PRECISION OF TWO METHODS OF MEASURING THREE WEIGHTS OF COTTON UNDERWEAR

Method	Lightweight		Medium-weight		Heavyweight	
	Wales	Courses	Wales	Courses	Wales	Courses
Bar	0.015	0.008	0.017	0.006	0.015	0.009
Flat	0.015	0.014	0.010	0.029	0.012	0.019
Ratio: $\frac{\text{Flat}}{\text{Bar}}$	1.00	1.75*	0.588	4.83*	0.80	2.17*

* Significant difference in precision.

TABLE V. COEFFICIENTS OF VARIATION IN MEASURING SHRINKAGE OF UNDERWEAR BY THE BAR METHOD

	Light-weight	Medium-weight	Heavy-weight
Wales	4.9	4.3	3.5
Courses	—	—	9.0

Table IV lists the pooled variances of the three measurements of the wales and four measurements of the courses on each of 10 laundered specimens using the bar and the flat methods. The ratios of the variances were utilized to test for differences in consistency between the methods. No significant difference exists between the two in measuring the wales, but the bar is significantly more precise in the measurements of the courses.

Table V lists the coefficients of variation of the average shrinkage measurements by the bar method. Since the average shrinkage measurements of the courses were less than 2 percent for the light- and medium-weight fabrics, regardless of operator or method, their variability was disregarded in these weights. However, the average shrinkage in the heavyweight fabric for the courses was − 6.2 percent for the bar method, with an average coefficient of variation of 9 percent. Computation of the sample size for estimating the average shrinkage of the courses is based on the variability of the heavyweight sample alone.

Using the procedures recommended by the American Society for Testing Materials,* it was deter-

* A.S.T.M. Standards on Textile Materials—Appendix VI, 1943.

mined that if the average wales shrinkages of 5 specimens were taken, the sample average would be within 5 percent of the true average 95 times in 100.

Owing to the greater variability in shrinkage measurements of the courses, it would be necessary to allow for an 8-percent tolerance limit if it were desirable to take 5 specimens with the same probability. It would be necessary to take 15 specimens in the latter case if a 5-percent tolerance limit were used.

It is apparent from the above discussion and analysis that a reliable method of evaluating the shrinkage of tubular knit goods may be had by substituting rotary-drying for oven-drying in the test procedure and utilizing a bar device to eliminate subjective measuring errors.

Acknowledgment

Thanks are expressed to Mr. Gerald Winston for his analysis of the statistical data and to Mr. Norman E. Roberts for his editorial assistance in the preparation of this report.

ABSTRACTS

SERVICES AVAILABLE

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A list of periodicals, the abbreviations used in references to articles, and the addresses of publishers of English language publications is given on page 37 of the January, 1947, issue.

Abstracts of Papers in *Textile Research Journal*, Volume 17, 1947

Walker Yarn Abrader

The median as a typical value for Walker yarn abrader data. Seaman J. Tanenhaus. TEXTILE RESEARCH JOURNAL **17**, 281–6 (May 1947).

The data obtained on the Walker yarn abrader in the determination of abrasion-resistance of yarns form frequency distributions which are decidedly positively skewed. The mean of such distributions, being affected by the extremes, tends to be atypical. The modal class is too broad for precise differentiation and the first breaks are too variable to be of use. The median, which is independent of the extreme values, is more representative. Furthermore, by computing the average median of several sets of yarns rather than determining the lot median, a saving of time of approximately 50% is effected. Author

Text. Research J. Nov. 1947

Uniform Abrasion Measurement

Solution of the problem of producing uniform abrasion and its application to the testing of textiles. Herbert F. Schiefer. TEXTILE RESEARCH JOURNAL **17**, 360–8 (July 1947); *J. Research Natl. Bur. Standards* **39**, 1–10 (July 1947).

A general mathematical solution to the problem of producing uniform abrasion over a plane area of a specimen from every azimuthal direction was worked out. This solution requires that the abradant and the specimen rotate in the same direction and with the same angular velocity. The specimen may revolve about the center of the abradant with any angular velocity in the same direction as or in the opposite direction from its direction of rotation. The specimen must not extend beyond the boundary of the abradant. Simple revolution of abradant about specimen and of specimen about abradant without rotation of either are special solutions. The special solution in which the specimen does not revolve about the center of the abradant is the simplest one from mechanical considerations. A machine based upon this special solution is described and preliminary results obtained with it are discussed. Author

Text. Research J. Nov. 1947

ABSTRACT REPRINTS

The JOURNAL *is again offering to its subscribers reprints of the Abstracts and Book Reviews sections, printed on one side of the sheet so that the items may be clipped, pasted on cards, and filed for permanent reference. Subscription to this service for 1948 is $3.00. Orders for this special service for 1948 should be mailed at once to Textile Research Institute, Inc., 10 East 40th Street, New York 16, N. Y.*

Air Permeability of Fabrics

Air permeability of fabrics. I. L. W. Rainard. TEXTILE RESEARCH JOURNAL **16**, 473–80 (Oct. 1946).

A plot of air flow divided by pressure differential against pressure differential gives generally a straight line whose two constants, the slope and the intercept at $\Delta p = 0$, namely,

the intrinsic permeability, described the properties of a fabric over a wide range. A method is also suggested for deriving some properties of fabrics with respect to air passage at low air velocities. The empirical equation described by this work is useful mainly in the very low to moderate pressure differential ranges, particularly in calculating the air flow through a fabric at any given pressure differential within the range. Author

Text. Research J. Nov. 1947

Air permeability of fabrics. II. L. W. Rainard. TEXTILE RESEARCH JOURNAL **17**, 167–70 (Mar. 1947).

The Hagenbach equation, which describes the streamlined flow of fluid through tubes with a correction for kinetic energy changes, may be applicable to the study of air flow through fabrics. An approximate indication of the application of this equation is obtained by determining the K_1 (slope) and K_2 (intercept) values for two and three layers of fabrics. The constants K_1 and K_2 are similar to the slope and the intrinsic permeability described previously and can also be used, but with a greater utility, in describing the air flow through a fabric for any given pressure differential up to the point of turbulence. However, K_1 and K_2 are two constants of a fabric which can be determined only experimentally at present and whose exact relationship to the number, length, and radius of the openings to the fabric is yet to be determined.

Text. Research J. Nov. 1947 Author

Air Permeability Measurements

Relationship between measurements of air permeability by two machines. M. I. Landsberg and Gerald Winston. TEXTILE RESEARCH JOURNAL **17**, 214–21 (Apr. 1947).

In this paper is described the derivation of the empirical relationship between two commonly used instruments for determining air permeability—namely, the Frazier and Gurley machines. In addition, the limitations of each of the devices are discussed as well as the number of specimens necessary for testing. The correlation has also been determined by consideration of physical constants and pressure differentials, using the empirical data obtained on the Frazier and Gurley instruments operating at a pressure of 0.5 and 1.26 in. of water, respectively. The equations derived were $\log Y_F = \log 533.0 - 1.02 \log X_G$, based upon the empirical data alone, and $\log Y_F = \log 507.5 - \log X_G$ when the physical constants of the machines were considered. Author

Text. Research J. Nov. 1947

Color and Chemical Structure

Relation of color perception to chemical structure: A critical bibliography. I. H. Godlove. TEXTILE RESEARCH JOURNAL **17**, 185–98 (Apr. 1947).

A critical selection of basic references (books and periodicals) intended to serve as a complete reference shelf on the subject. The triangular relationship of color perception, chemical structure, and physical data (typified by absorption spectra) is discussed and illustrated schematically. The references are classified in three sections with reference to a fourth (the Nickerson data), published previously in TEXTILE RESEARCH JOURNAL. A brief description of the scope of the publication accompanies each item.

Text. Research J. Nov. 1947

The Cotton Card

Working of the cotton card and its mathematical concepts. W. Oeser. TEXTILE RESEARCH JOURNAL **17**, 382–4 (July 1947).

The development of a method for the evaluation of carding intensity is traced. The measure suggested for use is the ratio of the number of wire points on the cylinder to the inches of fiber spread on the cylinder (wire points/inch of fiber length). The density of the wire clothing and the fineness of the raw material are considered in the evaluation.

Text. Research J. Nov. 1947 Author

Nitrogen Content of Cotton

Nitrogen content of cotton in relation to other fiber properties. Frank M. Eaton. TEXTILE RESEARCH JOURNAL **17**, 568–75 (Oct. 1947).

The percentage of nitrogen in American upland and Sea Island cottons is found to vary with the degree of fineness, maturity, and other physical properties of the fibers and their yarns. The correlation coefficients between percent nitrogen and these other properties were improved substantially when varietal and environmental influences were separated by covariance analyses. As examples of the significance or limitation of lint-nitrogen as an index value, the correlation coefficient for "varieties" between percent nitrogen and skein strength was 0.68 and between nitrogen and yarn appearance 0.52. Drought and changes in the carbohydrate and nitrogen content of the plant have notable influences on the nitrogen content of the lint.

Text. Research J. Nov. 1947 Author

Structure of Cotton Linters

Structure of cotton linters. Charles W. Hock. TEXTILE RESEARCH JOURNAL **17**, 423–30 (Aug. 1947).

The microscopic structure of cotton linters fibers is similar in many respects to that of staple (lint) cotton. There are no pronounced differences between these two types, although in general linters fibers are darker, shorter, more nearly cylindrical, and have thicker walls and narrower central canals than the staple cotton. The thin primary wall, which covers the surface of the fibers, consists largely of waxy and pectic materials. The thick secondary wall is arranged in layers. In some linters fibers this layered pattern is due to the alternation of layers of cellulose which differ in porosity and density, and in others to the alternation of layers of cellulose with layers of noncellulosic material. The width of individual layers varies roughly between 0.1 and 0.4 μ in unswollen fibers. The cellulosic layers are further subdivided into fine threadlike fibrils which make an acute angle with respect to the long axis of the fiber.

The orientation of the fibrils is not the same in all layers and may even reverse its direction of spiral in a single layer. These structural characteristics influence the manner of swelling. Author

Text. Research J. Nov. 1947

Diffusion of Water Vapor Through Textiles

Diffusion of water vapor through textiles. Lyman Fourt and Milton Harris. TEXTILE RESEARCH JOURNAL **17**, 256–63 (May 1947).

One factor influencing the suitability of fabrics for clothing is resistance to the passage of water vapor. Measurement of the low resistances of woven fabrics requires a different technique from that for the high resistances of waterproofing and packaging materials, since the resistances are close to that of air itself, which may be used as a convenient comparison standard. The resistance of a woven fabric depends on the kind of fiber, the thickness, and its tightness of weaving. With glass and vinyon, the resistance increases rapidly with density, showing that the main pathway through these materials is the air spaces. With cotton, viscose rayon, and wool, the tightest weaving produces resistances only four to eight times that of air, whereas those of ordinary fabrics range from two to four times that of air. This indicates that significant amounts of water vapor travel through the substance of the fibers themselves, which is confirmed by experiments on viscose film (cellophane). Nylon and cellulose acetate are intermediate. The effects of resistant fibers are seen when the fraction of the volume occupied by fiber exceeds 40%; below this level all fabrics are closely similar, since the low-resistance pathway through the air spaces is sufficiently available. Authors

Text. Research J. Nov. 1947

The Dyeometer

Studies on rayon dyeing: Applications of the Dyeometer. G. L. Royer, H. R. McCleary, and J. M. A. deBruyne. TEXTILE RESEARCH JOURNAL **17**, 438–47 (Aug. 1947); *J. Soc. Dyers and Colourists* **63**, 254–9 (Aug. 1947).

A quantitative study of rayon dyeing was made with a special apparatus, named the Dyeometer. The effects of manufacturing variations and variations in dyeing conditions on the dyeing characteristics of experimental and commercial rayon yarns, using Calcomine Sky Blue FF, were investigated. The equilibrium dye-bath exhaustion value was not affected by the presence of skin structure, by increase in filament size, godet stretch, or viscose age; it was slightly affected by the presence of delusterant (mineral oil, TiO_2); it increased with increase in D.P. of the viscose, size of the cation (Li^+, Na^+, K^+), and yarn weight; and it decreased with increase in dye-bath temperature and dye concentration. The dyeing rate increased with increase in NaCl concentration, size of cation of electrolyte, yarn weight, and dye-bath temperature; and it decreased with increase in filament denier, godet stretch, viscose age, D. P. of viscose, and with the presence of skin structure. Some practical aspects of the results are discussed. *Cf.* T. R. J. **16**, 616 (Dec. 1946). Authors

Text. Research J. Nov. 1947

Automatic Solar Exposure Cabinet

Automatic normal-incidence solar exposure cabinet. P. J. Fynn and K. S. Campbell. TEXTILE RESEARCH JOURNAL **17**, 158–66 (Mar. 1947).

Details of the mechanical construction and operation of a device designed for the controlled exposure of fabric samples to sunlight are given. A shallow cabinet, 4 ft. wide by 6 ft. long, housing the samples is mounted on a frame and geared to an automatic motor drive in such a way that the top of the cabinet remains normal to the sun's rays, within $\pm 3\frac{1}{4}°$, throughout the day. At sunset the cabinet is automatically returned to be in the correct position for sunrise the next morning. The top of the cabinet is fitted with glass filters of various types and an attachment is provided which records, once every 4 min., the intensity of the radiant energy incident upon the fabric sample directly beneath each filter. Means are furnished for controlling the temperature of the exposed samples and also for removing atmospheric contaminants from the air passing over them.

Text. Research J. Nov. 1947 Authors

Fiber Properties

Tensile and torsional properties of textile fibers. L. G. Ray, Jr. TEXTILE RESEARCH JOURNAL **17**, 1–6 (Jan. 1947).

In an effort to correlate single-fiber properties with fabric behavior, the compliance of fibers from several different polymers to tensile and torsional stresses was measured. For certain fibers, Young's modulus and the work hysteresis observed in subjecting the fiber to a cycle of elongation and relaxation in the range of 0.5 to 2.0% elongation are sufficient to indicate qualitatively the behavior of fabrics made from the fiber. In other cases, these properties are not sufficient to explain fabric behavior and it is postulated that fiber resistance to shearing deformations may have an influence on fabric properties. Measurements of torsional moduli of fibers are described and related in a general way to the forces between polymer chains. The ratio of Young's modulus to the torsional modulus of fibers is taken as a measure of mechanical anisotrophy which determines how a fiber will comply to a deforming force. Properties characteristic of wool-like and silklike fibers are indicated. Author

Text. Research J. Nov. 1947

Filling Power of Feathers

Investigation of methods for determining the filling power of feathers. Norman B. Edelman. TEXTILE RESEARCH JOURNAL **17**, 199–206 (Apr. 1947).

The study reported here is concerned with establishing the optimum method for determining the filling power of feathers and down mixtures both as a research tool and as an acceptance quality-control procedure. Typical load-compression curves of feathers and pressure-

distribution curves of a sleeper on a sleeping bag are shown. Author
Text. Research J. Nov. 1947

Friction of Running Yarn

Apparatus for determining the coefficient of friction of running yarn. Francis Breazeale. TEXTILE RESEARCH JOURNAL **17**, 27–31 (Jan. 1947).

An apparatus is described which allows the coefficient of friction of yarn against various materials to be read directly from a scale. A feature of the design is that the tension in the yarn, or the speed of yarn travel, does not influence the indicated value except in those cases in which these variables actually change the value of the coefficient of friction itself. Some typical results obtained with the assembly are shown. An increase in the coefficient of friction occurred with increase in thread speed at a constant pre-tension of 40 g., against smooth glass and stainless steel. There is a linear relationship between coefficient of friction and the logarithm of the tension in the thread at a constant speed of 100 m. per min., for glass and "Alsimag." The coefficient decreases with increased normal pressure of the yarn against the material under test.
Text. Research J. Nov. 1947

Measurement of Friction Between Fibers

Measurement of friction between single fibers. Nils Gralén and Bertil Olofsson. TEXTILE RESEARCH JOURNAL **17**, 488–96 (Sept. 1947).

A new apparatus for measuring friction between two fibers is described. The fibers are placed in horizontal positions at the desired angle to each other. One fiber is moved horizontally at a constant velocity. The other fiber is fixed in a horizontal balance of very small inertia and can be pressed against the first by means of loads on the balance pan. The balance bearing frame is fixed to a vertical torsional wire so that the torsional force will counteract the frictional force at every moment. A stick-slip movement will be obtained, where the points of slippage correspond to maximal (static) friction and the arithmetic mean of the points of slippage and brake correspond to the smaller dynamic friction that brakes up the slipping motion. The theories of the motion are given in the original paper. Some measurements on wool and artificial fibers are reported. The anti-scale values for wool are great and have a great dispersion; the with-scale values are small and have a small dispersion. The difference is decreased by treatment with bromine water. For smooth fibers the availability of the equation $F = \mu_0 N - \alpha S$ is demonstrated (F = frictional force, N = normal pressure, S = contact area, μ_0 and α = constants). It is shown that the dynamic friction is independent of the relative velocity of the fibers at the velocities used. The intended uses of the apparatus for studies on wool scaliness and frictional properties in drawing and spinning operations are discussed.
Text. Research J. Nov. 1947 Authors

Hydrolysis of Cellulose

Hydrolysis of native versus regenerated cellulose structures. O. A. Battista and S. Coppick. TEXTILE RESEARCH JOURNAL **17**, 419–22 (Aug. 1947).

Milder conditions of hydrolysis than have been heretofore generally used to characterize cellulosic structures chemically were employed for the purpose of comparing the hydrolytic accessibility of native and regenerated structures, respectively. It is assumed that the accessibility of the fine cellulosic structure is related to the rate of drop of the average degree of polymerization of the cellulose with time of treatment. Using 5N HCl at 18°C, the following samples of cellulose were evaluated: (1) cotton linters and acid-pretreated cotton linters, respectively, before and after nondegradative solution and regeneration; (2) cotton linters and acid-pretreated cotton linters, respectively, before and after swelling in 18% NaOH at 18°C; (3) viscose rayons made from cotton linters and acid-pretreated cotton linters, respectively. Authors
Text. Research J. Nov. 1947

Phase Microscopy

Application of phase microscopy to textile fiber research. H. Reumuth. TEXTILE RESEARCH JOURNAL **17**, 69–81 (Feb. 1947).

This is the translation of a manuscript obtained from the author by Werner von Bergen, Technical Intelligence Investigator in Germany for the Office of The Quartermaster General. Applications of the phase microscope to the study of textile fibers are discussed and illustrated with photomicrographs.
Text. Research J. Nov. 1947

The phase-contrast method and its applications in microscopy. A. Köhler and W. Loos. TEXTILE RESEARCH JOURNAL **17**, 82–95 (Feb. 1947).

This translation of a German paper describes the phase-contrast apparatus and discusses the theoretical basis upon which it operates. The fundamental differences between bright field and dark field and between positive and negative phase-contrast are discussed. Typical applications are illustrated.
Text. Research J. Nov. 1947

Textile Microscopy

The application of microscopy to the textile industry. G. L. Royer and C. Maresh. TEXTILE RESEARCH JOURNAL **17**, 477–87 (Sept. 1947).

The use of the microscope in the textile field is described, including the study of fiber morphology, fiber pretreatments, printing and finishing operations. The use of the polarizing microscope in determining the degree of orderly structure within the textile fiber is discussed as well as the application of the newer phase and electron microscopes. The materials used by the textile industry, such as dyes, finishing and delustering agents, were also studied. Fluorescence microscopy and optical staining, both useful methods of attack on textile problems, as well as photography,

all important for the recording of microscopic and macroscopic images both within and beyond the range of human vision and perception, are discussed. 53 references to the literature are given. Authors

Text. Research J. Nov. 1947

Protein Fibers

Natural and synthetic protein fibers. Milton Harris and Alfred E. Brown. TEXTILE RESEARCH JOURNAL **17**, 323–30 (June 1947); "Fibrous Proteins," Soc. Dyers and Colourists, 1946, pp. 203–6.

The structures of wool and silk are discussed, and the dependence of the mechanical properties of these protein fibers on their structures is analyzed. The importance of the cystine linkages in wool fibers in affecting mechanical properties is demonstrated from experiments in which increasing numbers of disulfide linkages are removed by reduction with thioglycollate and subsequent methylation. Mechanical properties in the wet state are markedly influenced by reduction in cystine content, whereas properties in the dry state are little affected, and reasons for this behavior are presented. On the basis of the analysis of the structures of the two natural protein fibers, it is shown that there are two distinct protein fiber models, the "wool model" and the "silk model." A discussion of the present synthetic protein fibers in terms of these models is presented. The mechanical properties of a wide variety of protein fibers were determined, and on the basis of these and other properties evidence is presented that the pattern for the production of synthetic protein fibers must be the "wool model." Tables are included in which the amino-acid compositions of wool and silk are recorded. Authors

Text. Research J. Nov. 1947

Radiant-Energy Reflectance

Radiant-energy reflectance of men's-wear colors. Helen F. Greenler and Frank J. O'Neil. TEXTILE RESEARCH JOURNAL **17**, 63–8 (Feb. 1947).

An investigation of the radiant-energy reflectance characteristics of men's-wear colors applied on worsted fabrics was made. The reflection of visible energy by fabrics was considered to be equally as important as the reflection of infrared energy in the determination of the comparative coolness of fabrics to be worn in sunlight. Average visible and infrared reflectance values and radiant-energy reflectances of various colors were obtained from spectrophotometric measurements made in the visible and near-infrared to 1,000 mμ. Light colors exhibited greater radiant-energy reflectance than medium or dark colors. In making a selection between two or more colors which appear equally light or equally dark, comparison of the infrared characteristics determines the cooler shade. In support of the data presented in the paper a practical application and results are described. Authors

Text. Research J. Nov. 1947

Resilience of Pile Fabrics

Notes on the resilience of pile floor coverings. O. P. Beckwith and J. L. Barach. TEXTILE RESEARCH JOURNAL **17**, 305–13 (June 1947).

The techniques used for a number of years by a carpet manufacturer in evaluating the resilience of pile floor coverings are described. The Schiefer compressometer was used. It was found that the most reliable test technique, in terms of predicting service performance, was to determine the work done in compressing the pile after the fabric pile had been subjected to repeated loadings on the Bureau of Standards type of carpet wear-test machine. The relationship between pile fabrics of different construction is shown in terms of the indices of work done. Pile fabrics made from various synthetic fibers and wool are compared. A general discussion of the need for compressional measurements on carpets is indicated and one method for determining existing loading conditions through the use of a wire strain gage and an oscilloscope is discussed. The results from a series of measurements are shown for walking on level floor and climbing up and down stairs. Authors

Text. Research J. Nov. 1947

Resilience of Textiles

Resilience of fibers and fabrics. J. H. Dillon. TEXTILE RESEARCH JOURNAL **17**, 207–13 (Apr. 1947).

This paper is a general discussion of the nature of the resilience of textiles. The various definitions of resilience found in the literature are discussed, and it is concluded that the most common and useful definition, the ratio of energy of retraction to energy of deformation, should be adopted. It is pointed out, however, that this definition will not always coincide with empirical ideas such as "hand." The various parameters upon which resilience depends are discussed in some detail and several quantitative expressions for resilience are given. Author

Text. Research J. Nov. 1947

Harold DeWitt Smith

Harold DeWitt Smith, his life and works. J. H. Dillon. TEXTILE RESEARCH JOURNAL **17**, 355–9 (July 1947).

An address honoring the memory of Harold DeWitt Smith, presented at the American Association for the Advancement of Science Conference on Textiles, New London, New Hampshire, July 10, 1947.

Text. Research J. Nov. 1947

Spectral Absorption Measurement of Cellulose Degradation

Absorption spectra in the detection of chemical changes in cellulose and cellulose derivatives. J. W. Rowen, C. M. Hunt, E. K. Plyler. TEXTILE RESEARCH JOURNAL **17**, 504–11 (Sept. 1947); *J. Research Natl. Bur. Standards* **39**, 133–40 (Aug. 1947).

The spectral absorption of thin films of cellulose acetate, regenerated cellulose, the acetate and regenerated cellulose after oxidation with nitrogen dioxide gas, and the regenerated cellulose after oxidation with periodic acid followed by chlorous acid are recorded for the infrared from 2 to 15 μ and the ultraviolet from 215 to 400 mμ. The regenerated cellulose absorbed ultraviolet radiant energy only at the short-wave-

length end of the region studied, and there the transmittance of a film 2.8 μ thick was only slightly reduced. The acetate and oxidized celluloses absorbed in this region to a greater extent but gave no narrow absorption bands. Marked changes were observed in some of the absorption bands in the infrared region in going from cellulose acetate to regenerated cellulose and to their oxidation products. These changes were correlated with changes in the OH, CO, and COOH groups. The results indicate the possibility of estimating the amount of these groups present by spectral absorption measurements.

Text. Research J. Nov. 1947

Denier and Strength Determination

Determination of denier and strength of single filaments by Vibroscope and Heim tensile tester. V. E. Gonsalves. TEXTILE RESEARCH JOURNAL **17**, 369–75 (July 1947).

The Vibroscope for determining density of filaments includes an audiofrequency generator, vibrator, and Zeiss binoculars. The theoretical basis of its operation is described. The Heim tensile tester used in Holland in connection with the Vibroscope is described in detail. Data obtained by use of the apparatus described are furnished.

Text. Research J. Nov. 1947

Tear-Strength Analysis

Analysis of tear strength. O. B. Hager, D. D. Gagliardi, and H. B. Walker. TEXTILE RESEARCH JOURNAL **17**, 376–81 (July 1947).

An analysis has been made of the factors involved in the A.S.T.M. Trapezoid Tear-Strength Test. The tear strength of a fabric measured by this test is dependent mainly on the extensibility of the threads, the breaking strength of the threads, the effective specimen length, and the number of threads per inch of fabric. An analytical interpretation of the part played by each of these factors resulted in a general equation relating tear strength to the breaking strength and extensibility of a given fabric. The evaluation of the derived equation gave a good correlation between the calculated and measured tear-strength values of the fabrics investigated.

Text. Research J. Nov. 1947 Authors

Stress Relaxation of Cords

Stress relaxation of cotton and rayon cords at constant length. Edward G. Burleigh and Helmut Wakeham. TEXTILE RESEARCH JOURNAL **17**, 245–55 (May 1947).

The Tobolsky-Eyring reaction-rate theory of elasto-viscous behavior has been applied to stress relaxation at constant length for cotton and rayon cords. The experimental data used in this application have been obtained by means of an apparatus employing the bonded metalectric strain gage for stress measurement without necessitating change in length of the cord. Results on stress relaxation at different conditions of humidity and temperature are reported. These data have been utilized in evaluating the molecular constants in the Tobolsky-Eyring theory by assuming that the flow unit in the cellulose fiber consists of multiples of the cellobiose unit. The resulting constants have permitted calculation of a theoretical tensile strength which more nearly agrees with fact than that based on primary bond strength. This suggests that the mechanism of fiber breakage is one involving slippage of the cellulose chains with respect to each other rather than the rupture of primary valence bonds. For the particular case of stress relaxation at 100% R.H. the process has been resolved into two components, the more rapid of which appears to be the limiting case in which cellulose chains are flowing as individuals. Authors

Text. Research J. Nov. 1947

Measurement of Swelling

Microscopical methods in the measurement of swelling in cotton fibers: A literature survey. Mary L. Rollins. TEXTILE RESEARCH JOURNAL **17**, 19–26 (Jan. 1947).

A critical discussion of the literature. It is shown that little work has been reported on the microscopical measurement of cotton fibers swollen in water. Several directions in which work is needed are pointed out. 28 references.

Text. Research J. Nov. 1947

Measurement of Cross-Sectional Swelling

Some comparative data on the cross-sectional swelling of textile fibers. Frederick F. Morehead. TEXTILE RESEARCH JOURNAL **17**, 96–8 (Feb. 1947).

A method of measuring the cross-sectional areas of swollen fibers from photomicrographs is presented. In the method described the comparison of areas of identical cross section eliminates the variable of fiber size and takes into account irregularities of fiber shape. Data are given on the cross-sectional swelling of various animal fibers, fibers from synthetic polymers, natural cellulose fibers, and the rayons.

Text. Research J. Nov. 1947

Tire Cords

Effect of alternate heating and cooling on the breaking strength and elongation of cotton and rayon tire cords. Walter S. Lewis. TEXTILE RESEARCH JOURNAL **17**, 431–7 (Aug. 1947).

Six different tire cords, including one mercerized and two unmercerized cottons, and three rayons, were subjected to alternate heating to 16 hr. at 265°F and then cooling for 8 hr. in a room temperature of 70°F and 65% R.H. The effects of these treatments on the tenacity (grams per denier) and elongation were determined at the end of 1, 24, 36, and 48 heating periods. Tests were conducted on the cords in the oven-dry state, and also after conditioning in a standard atmosphere of 65% R.H. at 70°F. Elongations at breaking load were read from the charts, measuring from the point where the pen first registers and also from the point where it leaves the axis. In all tenacity tests the mercerized cotton cord showed the greatest resistance to change, and was generally followed by one of the rayons. The unmercerized cotton cords gave better retention of tenac-

ity than the other two rayon cords under standard conditions of test, but this order was reversed under oven-dry conditions. All the rayons exhibited higher initial elongations and, with one exception, greater losses on heating and cooling than did the cottons. The two methods of measuring elongation gave a slightly different order of rankings of the cords. The degrees of polymerization of the cellulose changed after 48 heating and cooling cycles from 2,400 to 385 for Wilds 13 unmercerized cotton cord, from 2,360 to 310 for the Wilds 13 mercerized cotton cord, and from 430 to 129 for the rayon cords. Author

Text. Research J. Nov. 1947

Tension Tests on Tire Cords

The endurance of cotton tire cords and fibers under static tensioning. Enoch Karrer, James N. Grant, and Rollin S. Orr. TEXTILE RESEARCH JOURNAL **17**, 314–22 (June 1947).

As an aid in the further interpretation of tests of the endurance of tire cords under conditions of flexing, tests were made of endurance under static tensioning. The samples measured represented the following types of tire cord: rayon and cotton regular commercial cords; experimental cotton cords manufactured and mercerized at the Southern Regional Laboratory; and mercerized and unmercerized cotton cords from a selected variety manufactured on standard commercial equipment. The curves of relationship between the tensioning load and the logarithm of the time elapsing under tension before rupture were plotted from data obtained in typical observations made over a range of tensioning load of from 50–90% of the previously determined tensile strength of the cords. The relationship at room temperature was shown to be linear in cotton cords; but this simple logarithmic relation was not found in the rayon cords. In mercerized cotton cords at elevated temperature moisture equilibrium must be approached to obtain a linear relationship. Since the question arises as to whether rupture consists merely of the movement of fiber past fiber or of the movement of structural elements within the fiber, the endurance of single fibers under static tensioning was measured. The single fiber relationship was found to be very nearly that of the cords fabricated from the corresponding fibers. Rupture in the single fibers as well as in the cords made from the fibers appeared to be the terminal condition of a phenomenon of flow of the micro-elements of the fibers themselves rather than a phenomenon of movement of fiber past fiber or.

Text. Research J. Nov. 1947 Authors

Viscose Structure

Investigation of fine structure of freely coagulated viscose fibers by means of optical and electron microscope. M. Horio, K. Kobayashi, and T. Kondo. TEXTILE RESEARCH JOURNAL **17**, 264–80 (May 1947).

Cross and longitudinal sections of freely coagulated viscose fibers were examined by the optical and electron microscopes. The electron-microscopic observations revealed many features of the structure which have not been disclosed optically. The skin is formed regardless of drafting or stretching during spinning. The coagulation of viscose by the usual coagulating bath is a typical Liesegang effect giving alternate layers of different submicroscopic structure. This structure is correlated with phenomena which take place during filament formation and is discussed in relation to the action of regenerating and nonregenerating baths. The paper is illustrated with some unusual electron-microscope photographs. W. A. Sisson

Text. Research J. Nov. 1947

Information for Contributors

Information for contributors to *Textile Research Journal.* Anon. TEXTILE RESEARCH JOURNAL **17**, 287–9 (May 1947). (Reprints furnished on request.)

Style requirements of TEXTILE RESEARCH JOURNAL are outlined.*

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* Abstracts of the rest of the papers in this volume will appear in early issues.

ANALYSIS: TESTING: LABORATORY METHODS

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Maturity of Cotton

New dye technique shows maturity of cotton. Herbert O. Smith and Mary P. Barnett. *Textile World* **97**, 105–8, 201–2, 204, 206 (July 1947).

A technique of direct dyeing with a particular combination of two dyes of contrasting color and different dyeing properties is said to be very effective for the identification of thin-walled or immature cotton fibers in contrast to mature or thick-walled fibers. The fibers to be tested are dyed in a 40:1 dye liquor-to-fiber ratio at the boil which contains 1.2% Color Index #278 and 2.8% Chlorantine Fast Green BBL. After dyeing for 15 min. at the boil two additions of 2.5% common salt are made 15 min. apart in a total boiling time of 45 min. In order to increase the contrast in shades obtained by dyeing, a 30-sec. wash in boiling water is used which removes excess red but little of the green dye. Migration of the dyes during drying should be avoided. Colored photographs illustrate the contrasts obtained. The technique is said to be helpful in the study of nep-forming fibers as well as in determining the efficiency of blending mature and immature fibers.

J. A. Woodruff

Text. Research J. Nov. 1947

Analysis of Velan on Cotton

Some chemical aspects of the application of Velan PF to cotton fabrics. F. V. Davis. *J. Soc. Dyers and Colourists* **63**, 260–3 (Aug. 1947).

When cotton fabrics treated with Velan PF are hydrolyzed with 5% sulfuric acid and steam-distilled, formaldehyde is liberated which may be used to estimate the amount of Velan PF applied. Using the method described, pure stearamido-methyl pyridinium chloride gives a theoretical yield of formaldehyde. Stearamido-methyl pyridinium chlo-

ride (SAMPC) appears to be hydrolyzed by boiling water to stearic methylolamide, 1 g.-mol. of which yields 0.5 g.-mol. formaldehyde on acid hydrolysis, leaving a strongly water-repellent substance which, it is suggested, is distearamidomethane. When up to about 2% of SAMPC is applied to cotton, it appears to be almost entirely condensed with cellulose or in loose association with the cellulose; if the amount of SAMPC rises appreciably above 2%, some of it is present in the cured fabric as the dimeric ether of stearic methylolamide and as distearamidomethane. The chemically bound SAMPC may be estimated after extraction of the fabric with solvents for the decomposition products. Author

Text. Research J. Nov. 1947

Determination of "666"

Determination of hexachlorocyclohexane in impregnated cloth. Jerome Goldenson and Samuel Sass. *Anal. Chem.* **19**, 320–2 (May 1947).

Hexachlorocyclohexane ("666"), which has been found to be a powerful insecticide and miticide, is being tested in connection with a study of miticides for impregnating cloth. The object of the work reported here was to devise a method for determining "666" in the presence or absence of chlorinated paraffins which would be applicable to cloth impregnated with the insecticide and sufficiently rapid and simple to be performed by personnel with limited training. The procedure evolved consists in the extraction of an average sample of cloth by acetone in a Soxhlet apparatus, dehydrochlorination of the extracted "666" with 0.3*N* NaOH, and titration of the liberated chloride.

Text. Research J. Nov. 1947 Authors

Determination of Tetrahydro Naphthol

Determination of 1,2,3,4-tetrahydro-2-naphthol in impregnated clothing. Jerome Goldenson and Samuel Sass. *Anal. Chem.* **19**, 322–4 (May 1947).

This paper describes a method for the determination of the insect repellent 1,2,3,4-tetrahydro-2-naphthol in impregnated clothing, involving extraction of the compound from the cloth with ethyl ether, followed by acetylation with acetic anhydride in pyridine. The excess acetic anhydride is titrated in a pyridine-water solution electrometrically to a pH of 8.9, or, alternately, to the end point of a mixed indicator of phenolphthalein and thymol blue. Binders and emulsifiers, such as chlorinated paraffin, cellulose acetate, and Nacconol NR, do not have an interfering effect. For others which interfere slightly, such as polyvinyl alcohol and Tween 80, corrections may be made by running suitable blanks.

Text. Research J. Nov. 1947 Authors

Rapid Dialysis

A procedure for rapid dialysis. Anne Marie Biget. *Compt. rend.* **224**, 827–8 (1947) (through *Chem. Abstr.* **41**, 4694*b* (Aug. 10, 1947)).

A gel of cellulose acetate is prepared from a solution in saturated $Mg(ClO_4)_2$ containing 25–60 g./l. of the cellulose acetate. The gel is washed thoroughly, and is cut into pieces of uniform size and shape. A piece of the gel is immersed in the solution; the ratio of their volumes is about 3:2. After 15 min. it is removed, and a second piece of gel is added to the solution. In 6 cycles, 97% of a readily diffusible compound, such as NaCl, is removed from the solution. The extracted material is removed from the gel by methodical washing with a volume of solvent only a little greater than that of the original solution. Only 7.5 mg. of cellulose acetate dissolved from 800 g. gel in 1,000 g. water. The gel is not sensitive to mineral salts or to alcohol. The water can be replaced by alcohol, and the alcohol by C_6H_6 or Et_2O. It has a high resistance to solutions in the pH range 1–9; for a contact of 1 hr., the range is 0–11. With a mixture of starch, glucose, and NaCl, in 5 cycles of 15 min. each, 93% of the NaCl and 7% of the glucose were removed. Six additional cycles of 25 min. removed 6% of the NaCl and 15% of the glucose.

Text. Research J. Nov. 1947

Enzymes on Fibers

Influence of enzymes on textile fibers and their impurities. E. Jaag. *Chimia* **1**, 57–63 (1947) (through *Chem. Abstr.* **41**, 5312*b* (Aug. 20, 1947)).

The application of proteolytic enzymes as a preliminary laundry treatment was found to aid in removing from cotton fabrics a dried-in mixture of India ink, beef blood, and milk. The maximum effect was obtained at 35–40°C with 3–4 g./l. of Ferm.-Präp. B/1, higher concentrations giving no better results. Twelve repeated enzyme treatments for 2 hrs. each at 38°C had no detrimental effect on appearance, weight, or strength of either raw or bleached cotton or viscose rayon. Enzyme concentrations of 3, 9, and 27 g./l. caused losses in strength of, respectively, 29, 70, and 85%, and in weight of 10.6, 21.6, and 35.1% in wool. Silk and nylon lost only little in weight but the losses in strength were 10, 19, and 27% for raw silk, and 8, 12, and 23% for boiled-off silk. The fluidity of solutions of silk in copper-ethylenediamine was reduced by preliminary enzyme treatments. 9 references.

Text. Research J. Nov. 1947

Molecular Weight of Nylon

Determination of the molecular weight of nylon. J. E. Waltz and Guy B. Taylor. *Anal. Chem.* **19**, 448–50 (July 1947).

Methods for the determination of the molecular weight of nylon are described. The active end-groups of alcohol-soluble nylons are determined either potentiometrically or conductometrically. The amino end-groups of alcohol-soluble nylons may be determined by conductometric titration in the solvent phenol-alcohol-water. Carboxyl end-groups of the latter nylons are determined by dissolving the polymer in benzyl alcohol at 175°C and titrating with KOH, using phenolphthalein as an indicator.

Text. Research J. Nov. 1947 Authors

Plasticizers for Vinyl Resins

Survey of plasticizers for vinyl resins. M. C. Reed. *J. Polymer Sci.* **2**, 115–20 (Apr. 1947).

For satisfactory performance in vinyl chloride-acetate resins, the plasticizer must be compatible with the resin in a wide range of concentrations. Low volatility, low rate of extraction by oil and water, and good chemical stability are necessary in most applications. In special applications, good electrical properties, low flammability, preservation of compound flexibility at low temperatures, and freedom from odor and taste are also required. Physical test methods, suitable for the evaluation of plasticizers, are briefly described. Author

Text. Research J. Nov. 1947

Polyamides

Polymeric amides from diamines and dibasic acids. D. D. Coffman, G. J. Berchet, W. R. Peterson, and E. W. Spanagel. *J. Polymer Sci.* **2**, 306–13 (June 1947).

Methods for the preparation of fiber-forming polymeric amides from polymethylene diamines and polymethylene dicarboxylic acids and the stabilization of these polymers against viscosity changes are described. The variation of physical properties, particularly melting points, of the polymeric amides, with increasing chain length is discussed. Authors

Text. Research J. Nov. 1947

Multi-ingredient polyamides. W. E. Catlin, E. P. Czerwin, and R. H. Wiley. *J. Polymer Sci.* **2**, 412–19 (Aug. 1947).

Multi-ingredient polymeric amides have been prepared by the condensation, under amide-forming conditions of various combinations and ratios of components comprising hexamethylenediammonium adipate, hexamethylenediammonium sebacate, and ϵ-aminocaproic acid or ϵ-caprolactam. The physical properties of the investigated linear polymeric amides range, depending upon the polymer components and the proportions employed, from high-softening, stiff, and difficultly soluble compositions to relatively low-softening, pliable, and easily soluble polymers. Authors

Text. Research J. Nov. 1947

Polyesters

Influence of catalysts on polyester resins. J. Delmonte. *Plastics* (Chicago) **6**, 39–40 (Apr. 1947).

This paper gives data on gelling time and on appearance and strength properties of resins formed over a range of temperatures using various peroxide catalysts with a number of liquid resins of the type (usually unsaturated polyesters) used in preparing low-pressure laminates. It is suggested that the suitability of resins for use in laminates be determined by tests made on the resins in the absence of cloth reinforcement. W. E. Davis

Text. Research J. Nov. 1947

Diffusion of Water through Polymers

Diffusion of water vapor through high-polymeric membranes. A. E. Korvezee and E. A. J. Mol. *J. Polymer Sci.* **2**, 371–80 (Aug. 1947).

The water permeability of membranes of polyethylene, polystyrene, chlorinated polyvinyl chloride, cellulose triacetate, plasticized polyvinyl chloride, and a copolymer of vinyl chloride and vinylidine chloride at 25° and 50°C was determined. E. D. Klug

Text. Research J. Nov. 1947

Polymerization

Determination of the velocity coefficients for polymerization processes. I. The direct photopolymerization of vinyl acetate. G. M. Burnett and H. W. Melville. *Proc. Roy. Soc.* **A189**, 456–80 (June 3, 1947).

This paper describes a method whereby the individual velocity coefficients of a vinyl polymerization reaction have been determined by a detailed analysis of the kinetics of the photopolymerization of liquid vinyl acetate. The evaluation of the coefficients is made possible by the development of an accurate method for measuring the lifetime of the active polymeric molecules employing intermittent radiation. In addition, the kinetic chain length and the number-average molecular weight of the polymers have been measured. It is shown that each absorbed quantum of radiation activates one vinyl acetate molecule, probably as a diradical. The chain terminates by a disproportionation reaction between the polymer radicals. The size of the growth or propagation bimolecular coefficient decreases only slightly as the molecule grows, the energy of activation is 4,400 cal., and the steric factor of the growth reaction is 10^{-5}. In the termination reaction there is likewise only a small decrease of velocity coefficient with increasing molecular size—no energy of activation is required and the steric factor has the surprisingly large value of 10^{-2}. Authors

Text. Research J. Nov. 1947

Determination of the velocity coefficients for polymerization processes. II. The photosensitized polymerization of vinyl acetate by benzoyl peroxide. G. M. Burnett and H. W. Melville. *Proc. Roy. Soc.* **A189**, 481–93 (June 3, 1947).

The photopolymerization of vinyl acetate has been found to be very much accelerated by the presence of small quantities of benzoyl peroxide. This is considered to be the result of a photochemical decomposition of the peroxide yielding free radicals such as phenyl and benzoate. The dependence of the rate of polymerization on the peroxide concentration suggests that there is a considerable inhibition of the reaction due to the presence of the peroxide. The value of the intensity exponent approaches unity as the concentration of the peroxide is increased and the formal analysis of the reaction suggests that the inhibition is due to the peroxide

molecules themselves. The analysis of the induction period and the dark period shows that these are identical and therefore real, so that it can be concluded that they depend essentially on the nature of the initiation step. The value of the initiation velocity coefficient is calculated and the energy of activation for this step is found to be 8.4 k.cal./g.mol. Authors

Text. Research J. Nov. 1947

Determination of the velocity coefficients for polymerization processes. III. Effect of environment on polymerization processes. G. M. Burnett and H. W. Melville. *Proc. Roy Soc.* **A189**, 494–507 (June 3, 1947).

The kinetics of the photopolymerization of vinyl acetate in ethyl acetate and *n*-hexane solutions were investigated with a view to studying the effect of environment on the course of the reaction. The ethyl acetate solutions allow the polymer to remain extended so that the course of the reaction is similar to that of the pure monomer. With *n*-hexane, precipitation of the polymer takes place with the result that a considerable "gel" effect is observed. The reaction rate in both cases is proportional to the monomer concentration and to the square root of the light intensity. In ethyl acetate solutions the activation energy is 4.42 k.cal./g.mol., which is virtually the same as for the bulk reaction, while in normal hexane the value found is −1.30 k.cal./g.mol. Reasons for this have been suggested. The lifetime of the active polymer in *n*-hexane solution both before and after the inception of the gel stage has been measured using the sector technique, and the values of the coefficients for the propagation and termination reactions have been computed. It is found that the value of the former is little changed by the change in medium, while that of the termination step falls sharply—the fall being in agreement with the increase in rate. These results confirm present theories on the cause of the gel stage. Authors

Text. Research J. Nov. 1947

Osmotic Pressure in High Polymer Solutions

Osmotic pressure measurement and the instability of high polymers. A. F. Sirianni, L. M. Wise, and R. L. McIntosh. *Can. J. Research* **25B**, 301–21 (May 1947).

In attempting to obtain accurate osmotic data in the low-concentration range, it was found that the results were influenced by the diffusion of solvent from the cell through the membrane gasket. This behavior was prevented by auxiliary sealing in a cell of new design, and results obtained with the improved cell are compared with those obtained with previous types of osmometer. A partial re-examination of the influence of preparation of the membrane was made in order to check results reported by Robertson, McIntosh, and Grummitt, and the different behavior of caustic-swollen and water-swollen cellophane membranes has been confirmed, although quantitative agreement for water-swollen membranes between the two investigations was not obtained. Using caustic-treated membranes, the molecular weight of a polyvinyl acetate sample was found to be the same within experimental error in 3 solvents. Further results on the changes in intrinsic viscosity and molecular weight brought about by ageing at elevated temperatures in the presence and in the absence of oxygen are reported for polyvinyl acetate and polymethyl methacrylate. It appears that oxygen is necessary to bring about molecular-weight changes, and the degradation is due to scission of the chains at weak points. The evidence also suggests that changes in molecular shape may occur, since intrinsic viscosity values altered in some cases without evidence of molecular-weight variation. This last observation is apparently outside the limits of error in reproducing osmotic-pressure values. Authors

Text. Research J. Nov. 1947

Viscosity of High Polymers

The viscosity of macromolecular solutions. Yvon Conseiller. *Compt. rend.* **224**, 828–9 (1947) (through *Chem. Abstr.* **41**, 4694*g* (Aug. 10, 1947)).

The viscosity of macromolecular solutions was measured (1) with a capillary tube and (2) by placing them between 2 glass plates, applying a force to one plate, and measuring the rate of motion. Velocities of 10^{-2} to 10^{-5} c.g.s. units were convenient. Measurements were made on solutions of nitrocellulose in $PhNO_2$ and in ethyl phthalate, cellulose acetate in cyclohexanone, polyvinyl acetate in $PhCH_2OH$, and polystyrene in C_6H_6. There was a high initial rate of motion, followed by a decrease to a limiting value: the minimum ratio of the 2 velocities was 10. The movement was some thousandths of a mm. per min. The limiting viscosity was 70,000–4,600,000 poises. The time for establishing the limiting velocity is analogous to the time of relaxation: it was 1,300 sec. for a solution of nitrocellulose in $PhNO_2$. Evidently the high viscosity of macromolecular solutions does not preexist: it is developed by a deformation of the solution.

Text. Research J. Nov. 1947

Elastic Losses in High Polymers

Elastic losses in some high polymers as a function of frequency and temperature. H. S. Sack, J. Motz, H. L. Raub, and R. N. Work. *J. Applied Phys.* **18**, 450–6 (May 1947).

An attempt is made to apply the relaxation theory of elastic losses to experimental data on free and forced vibrations of specimens of some high polymers. Plasticized polyvinyl chloride, natural gum rubber, and various butadiene-styrene copolymer gum vulcanizates of varying content of butadiene to styrene are investigated. An apparatus for vibrating the test specimen, and 3 methods for measuring elastic losses are described. The specimen is mounted as a reed in such a way that during vibration it interrupts a light beam to a photocell and causes it to generate an A.C. signal proportional to the amplitude of

vibration. The effect of plasticizer content on the relationships of elastic modulus vs. temperature and of elastic losses vs. temperature for polyvinyl chloride is shown. Temperature dependence of the relationship of elastic loss vs. driving frequency is indicated for both natural and GR-S rubbers.
Text. Research J. Nov. 1947 T. J. Dietz

Infrared Spectroscopy

The study of macromolecules by infrared spectroscopy. H. W. Thompson. *J. Chem. Soc.* **1947**, 289–97 (Feb. 1947).

A review of the principles of the application of infrared spectroscopy to macromolecules, and some examples. Measurement of rate and mechanism of polymerization reactions, elucidation of the structure of molecules, derivation of facts likely to bear upon the order or arrangement of macromolecules in a given state of aggregation, study of changes caused by special treatments affecting the physical or chemical behavior of a macromolecule, and the general analysis of mixtures of macromolecules are discussed and applications and specific examples are cited. R. W. Eyler
Text. Research J. Nov. 1947

Viscosity-Concentration Coefficient

The effect of molecular-weight distribution on the reduced viscosity-concentration coefficient. W. E. Davis. *J. Am. Chem. Soc.* **69**, 1453–7 (June 1947).

Original data for cellulose nitrate and data from the literature for cellulose nitrate and polystyrene have been analyzed to show that the slope constant (K or K') in various equations connecting solution viscosity and concentration is a characteristic constant for a given solute-solvent system, and, in particular, is not appreciably affected by the molecular-weight distribution of the solute. Cases in which high values of the slope constant are obtained for cellulose nitrate and cellulose acetate can reasonably be attributed to variation in the chemical nature of samples which are in all cases examined either in unfractionated materials, head fractions, or fractions of very low molecular weight.
Text. Research J. Nov. 1947 Author

Stabilization of Wool

New developments in the chemical modification of wool. M. Harris and A. E. Brown. *Am. Dyestuff Reptr.* **36**, P316–19 (June 16, 1947).

A new method for stabilizing wool to the action of certain chemicals such as alkaline, oxidizing, and reducing agents has been developed. The method involves the conversion of the unstable disulfide cross-links to a more stable form by simultaneously rupturing the links and rebuilding them. This is accomplished by treating wool with a single solution containing a reducing agent such as sodium hydrosulfite and a rebuilding agent such as ethylene dibromide. In contrast to earlier work, which involved a long, impractical, and prohibitively expensive two-step method for modifying wool in this way, the new one-step method is rapid, simple, and inexpensive. It appears that unusual results may be obtained by application of these reactions to such industrial processes as stripping, bleaching, and vat dyeing.
Text. Research J. Nov. 1947 Authors

CHEMICAL AND PHYSICAL RESEARCH

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Acetylation of Cellulose

Course of acetylation and deacetylation reactions of cellulose fibers. I. Optical and swelling properties. D. Vermaas and P. H. Hermans. *J. Polymer Sci.* **2**, 397–405 (Aug. 1947).

Acetylations of model viscose filaments in ether-acetic anhydride-sulfuric acid with retention of the fiber structure were carried out to give a series of acetates of substitutions up to a maximum of 2.3. A higher degree of acetylation could not be obtained under the conditions employed without destruction of the fiber structure. Another series was prepared by deacetylating the acetates with aqueous ammonia. The birefringence and degree of swelling in water, acetone, and methanol for the acetylated and deacetylated fibers were determined. There is a slight hysteresis in the relative double-refraction curve. The plots of swelling vs. relative double-refraction for acetylated and deacetylated samples were quite different. This is said to be due to the greater accessibility of the amphorous region of the fiber during both acetylation and saponification.
Text. Research J. Nov. 1947 E. D Klug

Course of acetylation and deacetylation reactions of cellulose fibers. II. X-ray diagrams. D. Vermaas, P. H. Hermans, and A. Weidinger. *J. Polymer Sci.* **2**, 406–11 (Aug. 1947).

X-ray patterns for partly acetylated and deacetylated model viscose filaments (see preceding abstract) were obtained and the corresponding photometer curves are given. It is concluded that the rate of acetylation is greater in the intercrystalline areas than in the crystallites. Fibers acetylated to a substitution of 2.3, the maximum obtainable with retention of the fiber structure, gave the pattern identical with that of cellulose triacetate *II* after being recrystallized by heating in methanol or ethanol. The effect of recrystallization is to further increase the difference in the rate of saponification between the amorphous and the crystalline portions of the fiber. E. D. Klug
Text. Research J. Nov. 1947

Microbiological Degradation of Cotton

Fundamental aspects of the prevention of microbiological degradation of cotton textiles. R. G. H. Siu. *Am. Dyestuff Reptr.* **36**, P320–2 (June 16, 1947).

Of more than 1,000 cultures of microorganisms which have been

isolated from deteriorated cotton fabrics, about 200 have been shown to possess cellulolytic ability. In attacking the cotton the fungal hypha penetrates the fiber wall into the lumen, where it proliferates and digests the cotton fiber outward. Bacteria, on the other hand, adhere to the outer surface and pit their way inward. In both cases the attack appears to be a highly localized affair occurring only at the point of immediate contact with the microorganism. The organisms secrete cellulose-digesting enzymes. These have so far been classified into two classes, viz.: (*a*) cellulase, which converts cellulose into cellobiose, and (*b*) cellobiase, which converts cellobiose into glucose. Four general lines of approach to the development of preventive methods are (1) physical prevention of the organism from making contact with the cloth, as in the case of resin-impregnation, (2) cell toxicants, as exemplified by fungicides, (3) specific enzyme inhibitors, which exist today as theoretical possibilities, and (4) chemical modification of the cellulose molecule. Author

Text. Research J. Nov. 1947

Nitration of Cotton

Nitrations of swollen and collapsed cotton linters. Differences between the nitrogen contents and viscosities of technical and other nitrates prepared from highly swollen and collapsed cotton linters. R. K. Brown and C. B. Purves. *Pulp Paper Mag. Can.* **48**, 100–7 (1947) (through *Chem. Abstr.* **41**, 4917*b* (Aug. 10, 1947)).

One-half of each of 4 dry, dewaxed, highly swollen cotton linters samples had its accessibility reduced from a high value to about 0.25% by wetting in H_2O, followed by direct redrying at room temperature. Parallel nitrations with technical $HNO_3 = H_2SO_4 = H_2O$ mixtures yielded nitrates with about 12.2% N from the swollen linters, but the products from the collapsed linters contained 0.1–0.6% less. Results when the original linters, or the technical nitrates, were nitrated to the "trinitrate stage" (13.6–13.9% N) with $P_2O_5 - HNO_3$ were similar but less certain. The intrinsic viscosities in butyl acetate of corresponding nitrates from the swollen and collapsed nitrates were originally the same, but storage followed by reprecipitation from acetone introduced differences. In one case the collapsed linters gave technical nitrates and trinitrates whose solutions were visibly "grained" and of irreproducible viscosity. Although the swollen and collapsed linters certainly had the same chain-length distribution, fractionation of their "trinitrates" gave widely different integral distribution-intrinsic viscosity plots; that of the collapsed linters was at a somewhat higher viscosity level and had more points of flection. A similar but less pronounced difference existed between the plots for the renitrated technical nitrates, but those for the technical nitrates were nearly superposable. In the latter case, the somewhat higher viscosities, otherwise characteristic for plots of collapsed nitrates, appeared to be compensated by the effect of a lower N content in the short-chain range. The results suggest that chance variation in the technical drying of pulps may be reflected after nitration in products that fail to pass specifications in N content, viscosity, or solubility characteristics.

Text. Research J. Nov. 1947

Jute Fibers

X-ray diffraction study of dyed mercerized and intensively delignified jute fibers. N. G. Banerjee, B. S. Basak, and R. K. Sen. *Indian J. Phys.* **19**, 217–21 (1945) (through *Chem. Abstr.* **41**, 4925*c* (Aug. 10, 1947)).

Effects of the organic dyes methylene blue and Congo red on the structure of (1) raw jute fibers, (2) jute fibers subjected to intensive delignification, and (3) jute fibers subjected to incomplete mercerization were studied by means of x-rays. In intensive delignification it was found that the cellulose crystallites remain unaffected but their orientations parallel to the fiber axes become deranged considerably. This change did not take place on incomplete delignification. Thus it was concluded that a fraction of the lignin in jute cements the cellulose fibrils to parallelism in a jute fiber and gives strength to it. This part of the lignin is more difficult to remove. The dyes were found to produce no effect on either the structure or the orientation of the cellulose crystallites whether native or mercerized. No change could be discerned in the disheveled crystallite structure of intensively delignified jute.

Text. Research J. Nov. 1947

X-ray diffraction patterns of bleached jute fiber. S. C. Sirkar and S. K. Chowdhury. *Indian J. Phys.* **20**, 31–4 (1946) (through *Chem. Abstr.* **41**, 4925*f* (Aug. 10, 1947)).

The half widths of the 002, 020, and 120 reflections from jute fibers of 5 trade qualities were compared in the bleached and unbleached conditions. It was observed that the width of both 002 and 120 reflections becomes smaller and that of the 020 larger with bleaching in high-quality white, Tossa, and White top jute, but no change is observed with bleaching in low-quality White and Chinsura green jute. An explanation for this behavior based on chain lengths is given.

Text. Research J. Nov. 1947

Bleached Jute

The yellowing of bleached jute. P. B. Sarkar, H. Chatterjee, A. K. Mazumdar, and C. Nodder. *Current Sci.* **16**, 74–6 (1947) (through *Chem. Abstr.* **41**, 4925*g* (Aug. 10, 1947)).

Experiments with variously treated jute samples indicate that the hemicelluloses, which are not removed by treatment with $NaClO_2$, are largely responsible for the yellowing during storage. Residual lignin, lignin degradation products, degraded cellulose, and other noncellulosic impurities also contribute to yellowing. If steps are taken to remove the constituents that

cause yellowing, the fibers are weakened. It is likely that the best practical result that can be expected will be a product that is somewhat weaker than untreated jute but yellows only slightly.
Text. Research J. Nov. 1947

Theory of Plasticization

Application of a mechanistic theory of solvent action to plasticizers and plasticization. Arthur K. Doolittle. *J. Polymer Sci.* **2**, 121–41 (Apr. 1947).

The conventional thermodynamic theory of solvent action is briefly reviewed. A mechanistic theory is advanced to supplement the thermodynamic theory. The mechanistic theory views a resin solution as an example of dynamic equilibrium between the tendency of the solvent molecules to solvate the resin and the tendency of the resin macromolecules to unite with each other in three-dimensional aggregation. A plasticizer, being a nonvolatile solvent, remains in the resin film on evaporation of the solvent and thus opposes, to an extent depending on its concentration and solvent strength, the aggregation of the resin macromolecules. Although the permanence of plasticizers in resinous compositions does not correlate well with the author's measure of the relative solvent strengths of the plasticizers, very satisfactory correlation is obtained between the low-temperature behavior of plasticized films and the temperature dependence of the solvent strength of the plasticizers. Data are presented in support of the contention that a necessary condition for good low-temperature performance in plasticized resinous compositions requires that the plasticizer show a considerable improvement in solvent strength on cooling. Author
Text. Research J. Nov. 1947

Polyamide Fibers

Polycondensation of ω-*N*-methylaminohendecanoic acid. René Aelion and Georges Champetier. *Compt. rend.* **224**, 346–8 (1947) (through *Chem. Abstr.* **41**, 5311*d* (Aug. 20, 1947)).

The attractive forces between the >CO groups of one chain and the >NH groups of the adjacent chain in linear polyamide fibers are responsible for the resistance to chemical agents and for the elastic and plastic properties of these polymers (*cf.* Baker and Fuller, *C.A.* **36**, 6874[3], and Broser, Goldstein, and Kruger, *C.A.* **38**, 5714[1]). These forces are considerably weakened by the substitution of one H in the NH_2 group by a CH_3 group (Baker and Fuller, *C.A.* **37**, 4290[2]). This is verified in the polycondensate of ω-*N*-methylaminohendecanoic acid. The monomer, prepared by the treatment of hendecenoic acid with HBr gas and by the reaction of the brominated product with an alcohol CH_3NH_2 solution (Flaschenträger and Halle, *C.A.* **21**, 258), is polycondensed by heating it at the temperature of its melting point (134–135°C) in N atmosphere. The resulting polymer is opaque, whitish gray, melting at 60°C, can be spun into filaments which cannot be stretched easily, and is soluble in polar solvents such as glycol chlorohydrins and chlorinated hydrocarbons. The absorption spectrum in infrared is typical of disubstituted normal amines. The x-ray pattern of the nonoriented polymer is a diffuse ring corresponding to a spacing of 4.4Å.
Text. Research J. Nov. 1947

Compatibility of Polymers

Phase separation in polymer solution. A. Dobry and F. Boyer-Kawenoki. *J. Polymer Sci.* **2**, 90–100 (Feb. 1947).

The compatibilities of 14 high polymers (cellulose ethers and esters, polyvinyl alcohol and acetate, polystyrene, rubber and methylmethacrylate) dissolved in 13 solvents were determined. Of 35 pairs of high polymers tested, only four did not show separation; incompatibility appears to be the normal situation. With very few exceptions, 2 polymers, incompatible in one solvent, are incompatible in all solvents. As the molecular weight increases, the concentration of phase separation decreases. No obvious relationship between compatibility and chemical nature of the monomers was noted. Variations in temperature (0° to 55°C) had little effect on phase separation. A rough method of estimating molecular weights by comparing phase volumes is suggested. E. D. Klug
Text. Research J. Nov. 1947

Crystallization in High Polymers

Thermodynamics of crystallization in high polymers. I. Crystallization induced by stretching. Paul J. Flory. *J. Chem. Phys.* **15**, 397–408 (June 1947).

A theory of oriented crystallization in elongated polymers having network structures (e.g., in vulcanized rubber) is developed through the application of statistical mechanical procedures similar to those employed in the rubber elasticity theory. Expressions are derived which, within the limitation imposed by the simplifying assumptions, relate the incipient crystallization temperature with the elongation, the degree of crystallinity with the elongation and the temperature, and the retractive force at crystallization equilibrium with the elongation at constant temperature. The reciprocal of the absolute temperature for incipient crystallization is predicted to decrease linearly with a simple function of the elongation and the average number of chain segments between cross-linkages. Only moderate degrees of crystallinity are predicted at equilibrium. In conformity with requirements of the second law of thermodynamics, equilibrium crystallization decreases the tension in the stretched specimen. Apparent discrepancies between some of these predictions and various observations are attributed to severe departure from equilibrium crystallization when the polymer undergoes crystallization during the stretching process. A better approach to equilibrium should be achieved by stretching under conditions which prevent crystallization (e.g., at elevated temperature), then allowing crystallization to proceed at fixed elongation. Few experiments have

been performed in this manner, but such results as are available confirm, qualitatively, the predictions of the theory. Reasons for the rapid increase in tension which is observed when crystallization occurs during ordinary stretching of rubber are discussed. It is pointed out that crystalline and amorphous regions preferably should not be regarded as separate phases. Likewise, the conversion of amorphous to crystalline polymer does not conform to the definition of a phase transition.
Text. Research J. Nov. 1947 Author

Polymerization in Aqueous Solution

Polymerization of monomers in aqueous solution. M. G. Evans. *J. Chem. Soc.* **1947**, 266–74 (Feb. 1947).

Polymerizations in aqueous solution in the presence of ferrous ions and hydrogen peroxide were studied. The stoichiometry of the reaction between ferrous ions and hydrogen peroxide in the absence and presence of monomer, the kinetics of the hydrogen peroxide-ferrous ion reaction, the kinetics of polymerization, molecular weights and their distribution, end group determinations, and the dependence of the rate constants of certain of the reactions upon chain length are discussed.
Text. Research J. Nov. 1947 R. W. Eyler

Swelling of Polymers

Influence of structure on polymer-liquid interaction. I. Relative and absolute values of swelling equilibria. G. Salomon and G. J. Amerongen. *J. Polymer Sci.* **2**, 355–70 (Aug. 1947).

The swellings of vulcanized rubber, methyl rubber, Oppanol B–200, Buna S, Neoprene G, Perbunan, and Perbunan Extra were compared in series of the following types of solvents: (1) aliphatic hydrocarbons, (2) methyl alkyl ketones, (3) various types of ketones, (4) various types of unsaturated hydrocarbons, (5) simple aliphatics containing various types of polar groups, (6) benzene substitued with groups of different degrees of polarity, (7) organic bases, (8) methyl halides, (9) ethene polyhalides. The effect of polarity both of the polymer and of the liquid on swelling is illustrated by curves. The influence of temperature and of mechanical strain on the swelling of various elastomers is also discussed.
Text. Research J. Nov. 1947 E. D. Klug

Fractionation of Polyvinyl Acetate

Intrinsic viscosities and molecular weights of polyvinyl acetates. R. H. Wagner. *J. Polymer Sci.* **2**, 21–35 (Feb. 1947).

A medium viscosity polyvinyl acetate was fractionated. Intrinsic viscosities and osmotic-pressure molecular weights were obtained on the 15 fractions. From these data, the equation $[\eta]=(1.88\times10^{-4})M^{0.69}$ was obtained. Three of the fractions were refractionated and the equation for the second series of fractions was $[\eta]=(1.76\times10^{-4})M^{0.68}$. It follows that little improvement in homogeneity was effected by refractionation. A simple equation relating intrinsic viscosity to a single measured viscosity is given. The heterogeneity (ratio of viscosity-average to number-average molecular weight) of several samples was determined. The data indicate that heterogeneity increases with increase in molecular weight.
Text. Research J. Nov. 1947 E. D. Klug

Sodium Pectate

X-ray and moisture equilibrium investigations of sodium pectate. K. J. Palmer, T. M. Shaw, and M. Ballantyne. *J. Polymer Sci.* **2**, 318–28 (June 1947).

The variation of the x-ray reflection related to the interchain separation in sodium pectate has been determined as a function of moisture content. The crystalline regions in sodium pectate have been found to sorb about 24% water (dry basis) at a relative humidity of 95% at 25°C. The sample as a whole under these conditions has a water content of 64%. At high water contents, therefore, a large difference in water content must exist between the crystalline and amorphous regions of sodium pectate. A marked change in degree of crystallinity occurs, as determined by the number and sharpness of the x-ray reflections, at an average water content of about 6% on desorption, and on sorption of an average water content of about 14%. Authors
Text. Research J. Nov. 1947

Conductivity of Rubber

Electrical conductivity of GR-S and natural rubber stocks loaded with Shawinigan and R-40 blacks. P. E. Wack, R. L. Anthony, and E. Guth. *J. Applied Phys.* **18**, 456–69 (May 1947).

The resistivity of natural and GR-S rubber stocks, loaded with certain conducting blacks, is found to decrease with increasing concentration of black, to increase with increasing extension of the specimen, to decrease with time following a history of strain, and, in general, to increase with increasing temperature, at least for heavy loadings. The authors explain their results simply on the basis of the rupturing of conducting chains of carbon-black particles in the vulcanizates. Any disturbance which is likely to cause chain rupture may be expected to lead to increased resistivity. T. J. Dietz
Text. Research J. Nov. 1947

Sedimentation Equilibrium

A new optical method for observing sedimentation equilibrium. Gerson Kegeles. *J. Am. Chem. Soc.* **69**, 1302–5 (June 1947).

The theory for the use of a double prismatic cell for the simultaneous observation of the concentration distribution and concentration gradient distribution at sedimentation equilibrium has been presented. A cell and the experimental details connected with its use have been described. Results obtained in two experiments with purified ovalbumin have been encouraging and the method offers promise for accurate determinations of weight-average molecular weights in polydisperse systems. Authors
Text. Research J. Nov. 1947

BLEACHING: DYEING: FINISHING

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Coloration of Polyvinyl Chloride

Some problems connected with the coloration of polyvinyl chloride. James Haworth and B. N. Leyland. *Silk J. Rayon World* **23**, 30–2, 35–7, 52 (Feb., Mar. 1947).

The importance of color in extending the fields of application of polyvinyl chloride resins is reviewed briefly. Among the uses mentioned are plastic raincoats fabricated from unsupported calendered sheets, leather cloths, fabrics woven from monofilaments, and screen cloth. The compounding of typical resin mixes is described. Three methods of coloration at present available—namely, mass pigmentation, dyeing of the finished article, and selective copolymerization of vinyl monomers with color molecules—are discussed. The first has been most successful in industrial practice, the second having little commercial success to date, and the third being of academic interest only at present. The properties desirable in a pigment for use in vinyl resins include resistance to possible chemical effects of any or all of the ingredients of the mix; resistance to heat conditions in processing; no tendency to bleed from the mix or give rise to staining when mixes of different colors or types are in contact; and fastness to light and various other deteriorating conditions of service. Work was carried out on a range of Vulcafor colors, which are marketed for use in rubber compositions. Although in general these colors had the requisite heat resistance for use in vinyl resins, many were unsuitable owing to migration from the mix or to poor light-fastness in the new medium. Comparison of the relative light-fastness of pigments in rubber and in polyvinyl chloride indicates a consistent tendency towards inferior light-fastness in the latter medium. Organic pigments as a class seem to offer the best prospects for polyvinyl chloride, for they are brighter, stronger, and possess a greater variety of shades than inorganic pigments. The latter, while possessing certain desirable characteristics such as freedom from bleeding, are generally dull, weak, and flat in shade. The test for staining comprised storing pressed pigmented sheets in contact with white pressed specimens under a weight of 40 g. for 3 days at 50° ± 3°C and examining the white specimen for staining at the end of this period. Heat resistance was determined by storing colored specimens on glass at 170° ± 2°C for 30 min. with unpigmented controls and then observing shade changes against unheated specimens. For testing light-fastness, samples were exposed out-of-doors either to direct weather or under glass for 23 wks. The results showed no essential differences in the two methods of exposure. Preliminary work showed that none of the accelerated light-fastness tests correlated reliably with outdoor exposure. Results are reported showing the effects of different ingredients of a typical polyvinyl chloride compound on different classes of pigments. A list of colors considered suitable for most service demands is included.

Text. Research J. Nov. 1947 R. K. Worner

Dyeing Mixed Fabrics

Even dyeing of mixed fabrics. A. Ganthier. *Ind. textile* **62**, 57–8 (1945); *Chimie & industrie* **56**, 130 (1946) (through *Chem. Abstr.* **41**, 5310*i* (Aug. 20, 1947)).

To obtain an even shade on mixed fabrics it is necessary that the constituents of a mixture of leuco-esters of several vat dyes have a suitable affinity for the different fibers of the mixed fabric. In practice, such mixtures have not been used up to the present time. Perfectly even and very fast gray shades can be obtained on mixed fabrics by using mixtures of leuco-sulfuric esters of vat dyes containing: (*a*) a leuco-sulfuric ester of a vat dye that dyes cellulose fiber in a hot bath to give a shade varying from olive-gray to brown; (*b*) a leuco-sulfuric ester of dibenzanthrone or one of its substitution products giving blue shades, and (*c*) a leuco-sulfuric ester of an olive-green vat dye of given formula, or a 2,2-dialkoxybenzanthrone, which may be substituted and which gives green shades. Dyeing with these new mixtures can be carried out in the same way as for the leuco-sulfuric esters of vat dyes. The mixtures can be prepared directly in the dye baths, and other suitable leuco-sulfuric esters may be used.

Text. Research J. Nov. 1947

Dyeing Wool-Viscose Blends

Dyeing of yarn blends containing wool as one component. O. W. Clark. *Am. Dyestuff Reptr.* **36**, P269–74 (May 19, 1947).

34 photomicrographs are shown illustrating the comparative depth and penetration of dyeings of wool-viscose yarns dyed with vat dyes using 4 different formulas, azoic dyes, sulfur dyes, and soluble vat dyes. Detailed information on dyeing methods and a list of vat dyes suitable for viscose-wool mixtures are given in an appendix.

K. S. Campbell

Text. Research J. Nov. 1947

Union Dyeing

The dyeing of fast shades on piece goods where nylon and/or acetate are in mixtures containing wool. H. F. Clapham and G. R. Turner. *Am. Dyestuff Reptr.* **36**, P267–8, P274 (May 19, 1947).

Problems in dyeing wool-nylon, wool-acetate, and wool-nylon-acetate combinations are discussed. "Chromacyl" (premetalized)-type dyes are the most satisfactory for wool-nylon blends. "Blocking-off," in which one dye may partially or completely prevent the dyeing of a fiber by another dye, is generally unpredictable; it can be avoided by selecting dyes which dye at the same rate and penetrate to the same degree, or it may be overcome by adding another dye to the combina-

tion which has an effect opposite to the one causing the trouble. Wool-acetate blends should be dyed with selected acetate dyes which will stain the wool as little as possible and will have adequate fastness on the acetate, coupled with neutral-dyeing wool colors of satisfactory fastness to fill up the wool. Dyeing wool-nylon-acetate unions should be avoided at all costs.

K. S. Campbell

Text. Research J. Nov. 1947

Abbot-Cox Process

The Abbot-Cox process as applied to cotton. T. Richardson and E. R. Wiltshire. *J. Soc. Dyers and Colourists* **63**, 224–9 (July 1947).

A detailed description is given of the procedures of pigmentation, reduction, oxidation, and selection of vat dyes to be used in dyeing cotton in the package, ball warp, beam, loose form, and in the cop. Essentially the method consists of the exhaustion of carefully dispersed pigment by addition of electrolyte under selected temperature conditions. The reduction and oxidation processes are standard except that allowances are made for dyes whose exhaustion is affected severely by the dispersing agent used. 71 vat dyes are listed in a table describing the individual dyeing properties with respect to strike-test grading, sensitivity to the dispersing agent, time of reduction, and suitability for use in dyeing cotton.

J. A. Woodruff

Text. Research J. Nov. 1947

Chrome Dyes on Wool

Studies on wool dyeing: Comparison of methods for applying chrome dyes. G. L. Royer, H. E. Millson, and C. A. Amick. *J. Soc. Dyers and Colourists* **63**, 214–23 (July 1947).

When a wool staple is dyed with certain chrome dyes the tip portions are heavily dyed and the body portions are undyed or only lightly dyed. Cross-sectional examination shows that the tip portions of such a staple are heavily dyed, the body portions are undyed, and the intermediate portions are "ring" dyed, the thickness of the "rings" being greater toward the tip portions. This variation in dye penetration is caused by the condition of the epidermis of the wool fiber, and if this epidermal layer is broken or "damaged," the dye will penetrate the fiber more readily. When the dyed staple is carded or when wool is dyed after carding, the cross sections show unlevel distribution of dye from fiber to fiber. This fiber unlevelness causes poor color values, skittery or heathery effects, and entails an inefficient use of dye. Exhaustion curves show that there are variations in wools from different regions and after physical and chemical processing. These variations increase the possibility of fiber unlevelness when various wools are blended together. Wool exhibits reactivity in aqueous solutions and induces changes in pH both above and below the isoelectric zone—viz., pH 4–5. When a wool dye bath is made alkaline with ammonia at the start of the dyeing operation there is a gradual change in pH during the dyeing process which influences the chromation, the decomposition of dye by oxidation, and the exhaustion. Wool can be successfully dyed with chromable dyes which are not usually applied by the metachrome method. This can be accomplished by dyeing in a bath which initially has a pH greater than 7, by using an optimum low percentage of bichromate, by having magnesium sulfate present, and by employing a surface-active agent as a dyeing assistant. This procedure has been called the Calcomet process. The addition of magnesium sulfate to the alkaline dye bath results in the formation of a magnesium-dye complex without appreciably affecting the pH. The magnesium complex, however, appears to be unstable below a pH of about 7. The use of the specially designed surface-active agent as a dyeing assistant promotes dye exhaustion from the alkaline bath and also causes the exhaustion of any chromium-dye complex that is formed in the bath. Consequently, good penetration and levelness of shade are obtained. The afterchrome, chrome mordant, metachrome, and Calcomet methods were compared in the laboratory using several chrome dyes, and a detailed comparison of the various methods was made with Calcochrome Red ECB with respect to the rate of dyeing, the rate and extent of chromation, the pH, and the completeness of exhaustion.

Text. Research J. Nov. 1947 Authors

Sulfur Dyes

Sulfur-containing dyes from sodium derivatives and sulfur. H. Gault and M. Cogan. *Teintex* **11**, 20–1 (1946); *Chimie & industrie* **56**, 486 (1946) (through *Chem. Abstr.* **41**, 5310*e* (Aug. 20, 1947)).

By reaction of S on an alcohol solution of EtONa there is formed a coloring-matter that dyes silk. Aqueous solutions of such dyes are unstable, and S precipitates more or less rapidly when the solutions are allowed to stand in the air. When acidified they evolve H_2S with precipitation of a black, pulverulent mass containing S. From the similarity of the reactions NaOH + S and EtONa + S, it would seem that such dyes most probably consist of inorganic polysulfides, thiosulfates, or thionates. When fixed on the fiber, the dye is very stable, is unaffected by light or air, and but very slightly deteriorated by strong, concentrated acids. A number of other Na derivatives were studied from the standpoint of their ability to yield dyes with S: AmOH, glycerol, glycol, PhOH, mannitol, cyclohexanol, monoethanolamine, AcOH. The Ca derivatives are unsuitable for the preparation of such S dyes; only the Na derivatives (and presumably derivatives of other alkali metals) are suitable for this purpose.

Text. Research J. Nov. 1947

Action of Light on Dyes

Action of sunlight during dyeing process. V. D. Riswadkar. *In-*

dian Text. J. **56**, 1015–16 (Aug. 1946).

In dyeing Indanthrene Brilliant Orange R. K., direct sunlight was found to decompose the leuco compound, thus causing uneven dyeing effects. R. K. Worner
Text. Research J. Nov. 1947

Williams Unit

Williams Unit: Its use in textile processing. S. H. Williams. *Am. Dyestuff Reptr.* **36**, P256–62, P274 (May 19, 1947).

Practical suggestions concerning the use of the Williams Unit in various types of dyeing and textile processing are given and flow diagrams illustrating its utilization are presented. Developed primarily to fill a need for a small machine capable of dyeing a wide range of vat colors by a continuous process, it now finds use in direct, sulfur, and naphthol dyeing and, in addition, may be employed for highly efficient saturation (e.g., in a mercerizing range), and washing and scouring operations. Apparently, the success of the Williams Unit in dyeing is due in large part to the greatly increased dyeing action produced by the turbulence effected when cloth travels at high speed through very narrow channels in contact with the dye liquor. K. S. Campbell
Text. Research J. Nov. 1947

Chlorination of Wool

Felt-resistant wool by chlorination. H. Zimmerman. *Can. Text. J.* **64**, 41–4 (May 16, 1947).

The faults in processes dealing with chlorination of wool with sodium hypochlorite as the sole source of chlorine are discussed. A process is given whereby the hypochlorite is combined with selected sulfonamides and the combination applied to acidified wool over a period of approximately 45 min. It is shown that the exhaustion of chlorine to the wool is slower than by conventional methods and the rate may be controlled by the use of gradually rising temperature. Improved whiteness and hand, good strength, and abrasion-resistance are claimed. J. A. Woodruff
Text. Research J. Nov. 1947

Relation of Finishing to Fabric Construction

Dyeing, printing, and finishing in relation to fabric construction. R. S. Greenwood and C. P. Atkinson. *J. Soc. Dyers and Colourists* **63**, 208–13 (July 1947).

It is said to be essential that the detailed constitution of a fabric as well as its ultimate use should be known to the dyer, printer, or finisher in order that he may do his work intelligently. In a broad discussion, the variations in the physical and chemical properties of the fibers used, the yarn twists and counts, as well as the methods of yarn spinning, are all shown to require selected dyeing and finishing methods in order to obtain optimum results. The dyeing and finishing may be auxiliary to cloth design and construction, relying on yarn content and surface structure for their appeal. Again, fabrics may be designed to utilize the deliberate cooperation between the designer, manufacturer, and finisher. Another group of fabrics do not differ fundamentally in their finished state from the loom state, and are dependent upon the skill of the finisher rather than on cloth construction. Novel uses of Rayolanda and the soluble alginate yarns are given.
J. A. Woodruff
Text. Research J. Nov. 1947

Resin Finishing

The use of synthetic resins in the finishing of fabrics. D. Fornelli. *Am. Dyestuff Reptr.* **36**, 285–7, 312 (June 2, 1947).

Applications of synthetic resins in the field of fabric finishing are reviewed with particular attention to recent patents.
Text. Research J. Nov. 1947

Rotproofing

Use of naphthenates for the preservation of wood and fabrics. M. D. Gurwen. *Oil Colour Trades J.* **109**, 82–6 (1946); *Chimie & industrie* **56**, 122 (1946) (through *Chem. Abstr.* **41**, 5313*h* (Aug. 20, 1947)).

From observations made during the war, especially in New Guinea, where the climate is very hot and damp, Cu naphthenate proved to be the most effective protective agent against molds attacking ropes, fabrics, wood, etc. The product is difficult to obtain in solid form owing to the tendency to decompose during final drying. To overcome this difficulty a plastifier can be incorporated, or preferably the product can be prepared in the form of a double naphthenate of Cu and Al which can be obtained without decomposition and is less readily removed by water. Nets treated with the product retained 40% of their original strength after 17 months' immersion in sea water.
Text. Research J. Nov. 1947

Starch Substitute

Cereal starch vs. tamarind pectin as sizing material. N. S. Bhathena. *Indian Text J.* **57**, 548–9 (Mar. 1947).

Tamarind and some of the forest seeds powder have shown considerable promise as substitutes for cereal starches for textile sizing. Their use and further development for this purpose are urged in view of the acute food shortage in India.
Text. Research J. Nov. 1947 R. K. Worner

Evaluation of Water-Repellents

Evaluation of various water-repellent treatments on twenty-three cotton fabrics. Genevieve Smith and H. A. Wellington. *Rayon Text. Mo.* **28**, 332–4, 371–3, 423–5 (June, July, Aug. 1947).

The study was planned to determine the most desirable concentration of water-repellent, the best method of application, and the durability of home-applied water-repellents. The fabrics used were cotton drills, denims, jeans, and twills. The 3 water-repellents used were nondurable types. The results of laundering, spraying, and pressing tests show that the durability depended considerably on the type of water-

repellent and the concentration of application. J. A. Woodruff
Text. Research J. Nov. 1947

FIBERS: YARNS: FABRICS: MECHANICAL PROCESSES

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Fiberglas

Fiberglas yarns for textile purposes. Anon. *Can. Text. J.* **64**, 36–8 (Apr. 4, 1947).

Fundamental technical data on the manufacture of Fiberglas yarns, their physical properties, yarn-number conversion tables, the Fiberglas numbering system, and the technique of warping, beaming, and weaving are given. Author
Text. Research J. Nov. 1947

Nylon from Furfural

Nylon from corncobs. S. D. Kirkpatrick. *Chem. Eng.* **54**, 100–1 (Apr. 1947).

A new process for synthesizing raw materials for nylon production from furfural, which may be obtained from such materials as corncobs and oat hulls, is described. A flow sheet illustrates the process involving conversion of furfural to furan, of furan to tetrahydrofuran, of tetrahydrofuran to dichlorobutane, and of the latter to adiponitrile. Both hexamethylene diamine and adipic acid may be produced from the adiponitrile. Some experimental details are given. R. W. Eyler
Text. Research J. Nov. 1947

Protein Fibers

Structure of protein fibers. F. R. Senti. *Am. Dyestuff Reptr.* **36**, P230–7 (May 5, 1947).

Chemical structure, x-ray diffraction patterns, fine structure as shown by electron micrographs (of collagen), and load-elongation curves are discussed in relation to the physical properties of natural and synthetic protein fibers. It is pointed out that at low elongations casein fiber is more wool-like than silklike in its properties, showing a high degree of work recovery. In contrast to silk and wool, which possess good strength when wet, the artificial protein fiber as yet is of very low crystallinity and must be treated chemically to provide cross-linkages between molecular chains and build up wet strength to some degree.
K. S. Campbell
Text. Research J. Nov. 1947

Preparation and Properties of Staple Fiber

Influence of methods of preparation on the properties of pulp and staple fiber. R. E. Dörr. *Reichsamt Wirtschaftsausbau* Prüf-Nr. **54** (PB 52006), 29–39 (1940); cf. *C.A.* **34**, 3078[7]; **35**, 620[3]; **36**, 6012[6] (through *Chem. Abstr.* **41**, 5301*d* (Aug. 20, 1947)).

By the use of a series of bar graphs, Dörr shows the effects of various acid or alkaline treatments on the α-, β-, and γ-cellulose, and the glucose, mannose, and xylose contents of spruce, beech, pine, aspen, and straw pulps. (Invariably, small amounts of mannose-yielding materials are reported in the hardwoods.) Whereas repeated alkali extraction gives a pulp nearly free from hemicelluloses, the residual α-celluloses still retain appreciable amounts of xylan and small amounts of mannan. Too high a xylan content gives rise to marked difficulties in viscose filtration, and xylan and mannan may have an influence on the tensile strength and uses of the staple fiber (*I*). This was shown quantitatively by a correlation between total xylan and mannan content of the pulp and the dry strength of *I*. On the other hand, the higher the actual glucose content of the pulp, the greater was the tensile strength of *I*. This relation held true for commercial samples of *I*, the highest tensile strengths being reached when the fiber (on hydrolysis) gave 96–7% glucose.
Text. Research J. Nov. 1947

Frictional Properties of Wool

The frictional properties of wool and other textile fibers. E. H. Mercer and K. R. Makinson. *J. Text. Inst.* **38**, T227–40 (May 1947).

Diagrams are given of the stick-slip apparatus used in this study. With loads within the range of 0.01–0.02 g.wt., and under the conditions studied, the coefficient of friction was found to increase as the load decreased. It is said that the coefficient of friction measured from root to tip and from tip to root as well as the frictional difference and the relative frictional difference should be studied, although it should be borne in mind that neither of these functions may be related directly to the feltability of the mass of fibers. In connection with the variation of friction with pH the use of clean fibers is discussed. Special conditions are said to be necessary for buffer contact without emulsification when contaminated fibers are used. The importance of the use of clean fibers is also demonstrated in testing the variation of friction with temperature. The coefficients of friction previously mentioned are found to follow the results expected from changes in the handle of wool treated with alcoholic caustic potash and then with softeners. Considerable theoretical discussion is given in an attempt to show that the frictional difference of the wool fiber must be due to a ratchet effect. 21 references. J. A. Woodruff
Text. Research J. Nov. 1947

Worsted Drawing

Worsted drawing advances. Howard Martin. *Text. Mercury and Argus* **117**, 308 (Sept. 5, 1947).

The newly developed American system of spinning, employing modified cotton machinery, is summarized with respect to raw materials, processes, mechanical data, and yarn-count limitations. Recently developed apparatus, resulting in the effective measurement of sliver and yarn variation, has shown that the number of worsted open drawing operations necessary for fine roving can be reduced from 9 and 10 operations to 5 or 6 processes without affecting yarn quality or levelness.
Text. Research J. Nov. 1947 L. A. Fiori

Torsion in Cylinders

Torsion of a rubber cylinder. R. S. Rivlin. *J. Applied Phys.* **18**, 444–9 (May 1947).

Experimental confirmation is supplied for the theory that to produce pure torsion on a right circular cylinder of an isotropic, incompressible, highly elastic material, such as pure gum Hevea rubber, force must be applied normally to the plane ends of the cylinder.

Text. Research J. Nov. 1947 T. J. Dietz

Beam Warping

Beam warping. John H. Strong. *Text. Mercury and Argus* **117**, 225–7 (Aug. 29, 1947).

Suggestions are made for the improvement of beam warping. Setting of the creel in its relation to the back reed, reed wire construction for the elimination of thread strain, and specific adjustments for the efficient operation of the falling rollers are given. In addition, mention is made of the various reasons responsible for faulty beaming.

Text. Research J. Nov. 1947 L. A. Fiori

O. T. S. REPORTS*

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Fireproofing of Fabrics

Fireproofing of fabrics. J. E. Ramsbottom. (British Commonwealth Scientific Office Rept. 109–B1–2.) PB L 78693, 1947; 124 pp.; microfilm, $3.00—photostat, $9.00 (through *Bib. Sci. and Ind. Reports* **6**, 547 (Aug. 8, 1947)).

In the second report of the Fabrics Research Committee issued in 1930, an account was given of experimental work on fireproofing of fabric which had been in progress at the Royal Aircraft Est. This present report carries these investigations further and an attempt has been made to give a comprehensive summary of the work from its inception and to include the latest information available. Although the work has been concerned mainly with fabric, it has been thought desirable to include the results of the investigations on the fireproofing of Kapok and fireproof finishes for airplanes, which are closely related problems. The report contains a number of tables and graphs as well as an Appendix giving a complete list of fireproofing processes.

Text. Research J. Nov. 1947

German Linen Industry

Report on linen industry in Germany. T. H. McClaren and others. (BIOS Final Rept. 1218, Item 22, 31.) PB L 78648, Mar. 1947; 184 pp.; microfilm, $4.00—photostat, $13.00 (through *Bib. Sci. and Ind. Reports* **6**, 547 (Aug. 8, 1947)).

This is a complete and final report. Part I is a general review covering 41 plants visited and investigated. Part II covers the following subjects: flax production investigated in 3 plants; Flockenbast ("cottonized" flax) investigated in 4 plants; spinning, investigated in 7 plants; weaving, investigated in 14 plants; and bleaching, dyeing, and finishing, investigated in 13 plants. Appendix I contains staple analysis of Flockenbast, with table and illustration, and Appendix II contains analyses of samples of yarn, thread, and cloth, with tables. 11 illustrations are also included.

Text. Research J. Nov. 1947

German Manufacture of Acetate Rayon

Materials and machines used in the manufacture of cellulose acetate rayon. H. Livesey and E. W. Fletcher. (BIOS Final Rept. 1133, Item 22, 31.) PB L 77730, Sept.-Oct. 1946; 74 pp.; microfilm, $2.00—photostat, $5.00 (through *Bib. Sci. and Ind. Reports* **6**, 547 (Aug. 8, 1947)).

This document reports results of a study by a team of British investigators of materials and machines used in the manufacture of cellulose acetate rayon in Germany. It is based on interrogation of German leaders in this field and visits to a number of plants. It deals especially with the manufacture of ceramic thread guides, roller-bearings inserts, gear pumps used in rayon industry, sicromal tubing and welding rods for bronze vessels for the manufacture of cellulose acetate. In addition, processes covering the German manufacture of cellulose acetate and cellulose acetate rayon are fully described. Drawings are included.

Text. Research J. Nov. 1947

German Rayon Machinery

Report on machinery for the manufacture of viscose and spinning of continuous filament and staple fiber yarn in Germany. R. A. Gillespy. (BIOS Final Rept. 454, Item 31.) PB L 77709, Oct. 1945; 31 pp.; microfilm, $1.00—photostat, $3.00 (through *Bib. Sci. and Ind. Reports* **6**, 547 (Aug. 8, 1947)).

This report describes the machinery for the manufacture of viscose and spinning of continuous filament and staple fiber yarn used in 8 large German factories. Drawings and photographs are included.

Text. Research J. Nov. 1947

Stiffness Evaluation

A study of methods suitable for the evaluation of the stiffness and resilience of coated and impregnated gasproof cloth. With Appendix I. Graphs nos. 1–276. Irving Pockel and Otto Morningstar. (Mass. Institute of Technology Memo Rept. 30.) PB L 78637, Apr. 1943; 310 pp.; microfilm, $7.00—photostat, $21.00 (through *Bib. Sci. and Ind. Reports* **6**, 547 (Aug. 8, 1947)).

The object of the work described in this report was to develop a reliable method for the determination of the relative stiffness and resilience of coated and impregnated gasproof cloth. Four methods for the determination of the relative stiffness and resiliency of coated fabrics have been considered. The ring-loop method proved to be the most reli-

* Copies of these reports may be obtained directly from the Office of Technical Services, Department of Commerce, Washington, D. C. Orders should be accompanied by check or money order payable to the Treasurer of the United States.

able. The changes of stiffness and resilience of coated fabrics with respect to temperature were evaluated by the ring-loop method at various temperatures from 70°F to −40°F. Drawings and photographs are included. Appendix I presents graphical data. For Appendix II, see PB L 78636.
Text. Research J. Nov. 1947

MISCELLANEOUS

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Copper Number

A simpler determination of copper number of pulp. Shinichi Honda and Kazuo Hagiwara. *J. Agr. Chem. Soc. Japan* **17**, 447–57 (1941) (through *Chem. Abstr.* **41**, 4919*a* (Aug. 10, 1947)).

By the use of the Schwalbe-Braidy method as improved by Clibbens-Geake and the following reagents proposed by Honda and Hagiwara, the Cu number of pulp can be determined with a relatively small error: 10% $CuSO_4 \cdot 5H_2O$; 350 g. $Na_2CO_3 \cdot 10H_2O$, and 50 g. $NaHCO_3$ in 1 l. water; 100 g. $Na_2MoO_4 \cdot 2H_2O$, 75 cc. of 83% H_3PO_4, and 275 cc. of concentrated H_2SO_4 in 1.75 l.; 0.05 *N* $KMnO_4$; 10% Na_2CO_3 for washing and asbestos for plugging filter funnels.
Text. Research J. Nov. 1947

Cotton in Bituminous Construction

Cotton fabrics in bituminous construction. Paul F. Critz. *Public Roads* **24**, 251–60 (Oct.-Nov.-Dec. 1946).

This article reports comprehensively an experiment begun in 1935 by the Public Roads Administration to investigate the feasibility of using cotton fabric in low-cost bituminous road surfacing. It is concluded that use of cotton fabrics increased the cost of construction but generally produced no important benefits.
Text. Research J. Nov. 1947 R. B. Evans

Spooner Drying Machine

The Spooner vertical drying machine. Anon. *Dyer* **98**, 177–8 (Aug. 15, 1947).

The construction features of this machine are discussed. Emphasis is placed on the sectional construction, the system of air circulation, and the claim that maximum shrinkage of fabrics is allowed in drying.
J. A. Woodruff
Text. Research J. Nov. 1947

Dye Nomenclature

Dye nomenclature: A plea for more system. Wm. H. Cady. *Am. Dyestuff Reptr.* **36**, 253–4, 279 (May 19, 1947).

Inconsistencies and inadequacies in the present system of commercial names for dyes are itemized and suggestions for improving the situation are made.
Text. Research J. Nov. 1947

Leather Technology

Industrial and apparel leathers. H. B. Whiting. *Am. Dyestuff Reptr.* **36**, P298–302 (June 2, 1947).

The various processes entailed in the manufacture of leather (curing, tanning, dyeing, finishing, etc.) are briefly described.
Text. Research J. Nov. 1947

Modified Nylon

Use of formaldehyde for modifying nylon. A. J. Hall. *Silk J. Rayon. World* **23**, 30–2; **24**, 55, 57, 59 (Apr., June 1947).

A review.
Text. Research J. Nov. 1947

Organo-Silicon Compounds

Organo-silicon compounds. H. G. Emblem. *Dyer* **98**, 221–2 (Aug. 29, 1947).

The potential uses in the textile industry of these compounds in promoting slip-resistance and water-repellency are given. J. A. Woodruff
Text. Research J. Nov. 1947

Pectin

Advances in pectin chemistry. Part I. Rudolph Speiser. *J. Polymer Sci.* **2**, 281–9 (June 1947).

A review of recent work on the constitution and structure of pectin, the characterization of pectin, x-ray diffraction studies on sodium pectate, and the acid dissociation of pectinic acids. E. D. Klug
Text. Research J. Nov. 1947

Structure of High Polymers

The study of high-polymer structure by x-ray diffraction methods. C. W. Bunn. *J. Chem. Soc.* **1947**, 297–306 (Feb. 1947).

A review of the evidence regarding structure that is obtainable by x-ray diffraction methods. Examples of such studies are explained. 19 references. R. W. Eyler
Text. Research J. Nov. 1947

Peanut Protein Fiber

Peanut protein fiber: Its position in the protein fiber world. Walter M. Scott. *Chemurgic Digest* **6**, 192–5 (June 30, 1947).

Preparation of peanut protein and methods for its manufacture into fiber, as developed at the Southern Regional Laboratory, are discussed. Protein fibers can be produced from several sources of industrial protein, including casein and meals from soybeans, peanuts, and cottonseed. All of these materials are by-products which, considering present uses, may not prove to be adequate sources of raw material for protein fibers. R. B. Evans
Text. Research J. Nov. 1947

Yucca Fibers

Yucca fibers. C. W. Botkin. *Chemurgic Papers*, 1947 Series, No. 4. Columbus, O., National Farm Chemurgic Council. 6 pp.

Yucca fiber was used by aborigines of the Southwest for sandals, shrouds, royal robes, etc., and 80,000,000 lb. were used during World War I for cotton bagging. Botanical classification, fiber yield, harvesting and fiber separation methods, and properties are discussed. The fiber is

reported to be strong enough for binder twine, rope, and wrapping twine. It may also be used for laminated plastics, pads, insulation, and brushes. R. B. Evans
Text. Research J. Nov. 1947

Ramie

Ramie: 1947. Earle L. Rauber. *Monthly Rev. Federal Reserve Bank, Atlanta* **32**, 81–6 (July 31, 1947).

Ramie was used during the war as stern-tube packing for ocean-going vessels in the United States and for parachute harness in Canada and Great Britain. Potential uses are discussed and it is reported that considerable shipments are now being made to Switzerland, Italy, and France. There are now substantial plantings of ramie in the Everglades and in Orange County, Florida, and smaller acreages in Alabama, Louisiana, Mississippi, Texas, and, more recently, California. Concerns involved are named and discussed comprehensively. It is reported that one of these is approaching or has reached profitable operation. Research on ramie is being conducted by the Office of Technical Services, the U. S. Dept. of Commerce, the U. S. Dept. of Agriculture, and the State of Florida, as well as by private concerns. The industry is said to need lower growing and processing costs and better coordination between different branches of the industry; technical obstacles no longer seem insurmountable.
Text. Research J. Nov. 1947 R. B. Evans

Porosity of Fibers

The porosity of textile fibers. J. W. Illingworth. *Silk J. Rayon World* **23**, 28–9; **24**, 50, 52, 59 (Mar., June 1947).

A résumé of the literature, with references.
Text. Research J. Nov. 1947

Acrylic Resins

New process for acrylic resins. Richard W. Porter. *Chem. Eng.* **54**, 102–6 (Apr. 1947).

A detailed description of a new process for manufacture of methyl methacrylate monomer, molding powder, and transparent sheet. Basic raw materials used in producing the monomer are calcium carbide, acetone, and methanol. Other materials used are potassium hydroxide, phosphorus pentoxide, carbon dioxide, and solvents. 4 flow sheets are included. R. W. Eyler
Text. Research J. Nov. 1947

Termites, Microorganisms, and Fungi

Attack by termites, microorganisms, and fungus on cotton fibers. Narhari H. Shah. *Indian Text. J.* **57**, 241–50 (Dec. 1946).

A review. Work on fungicides during World War II, a practical experiment on the use of copper with chromium and iron on mineral khaki for producing an effective fungicide and bactericide, and a study of the effectiveness against termite and microorganism attack of 16 finishing treatments applied to osnaburg are described.
Text. Research J. Nov. 1947 R. K. Worner

Terylene

Terylene — New all-British fiber marks important advance in the synthetic field. J. Gordon Cook. *Silk J. Rayon World* **23**, 28–30 (Nov. 1946).

The development of the new fiber Terylene and some of its properties are described briefly. Terylene is a polyester resulting from the condensation of terephthalic acid with ethylene glycol. In its present developmental stage, properties indicated include launderability; ability to be heat-set; good resilience; high ratio of wet-to-dry strength; resistance to microorganisms and bacteria, to chemicals such as acids, organic solvents, and bleaching agents, and to heat and light; high initial elastic modulus; and low moisture absorption.
Text. Research J. Nov. 1947 R. K. Worner

A long-awaited survey

FLAMEPROOFING TEXTILE FABRICS

Prepared by a Staff of Specialists under the Editorship of ROBERT W. LITTLE, Major, Q.M.C., under Research Project Q.M.C. #27 of the National Research Council, National Academy of Sciences.

Office of the Quartermaster General, Military Planning Division, Research and Development Branch

American Chemical Society Monograph No. 104

This new volume on the flameproofing of textile fabrics is based on problems encountered during the war. It is divided into three general sections: (1) the fundamental mechanisms of the thermal degradation of cellulose and the chemical or physical phenomena of flameproofing; (2) the methods employed in the processing and evaluation of flameproofed fabrics; and (3) the various applications for flame-retarding treatments in the field of textile fabrics. This presentation familiarizes the reader with the types of retardants available, the function of these agents in the prevention of flaming and glowing, and the methods by which they are applied and evaluated. In addition to the results of the research project on "The Flameproofing of Army Clothing," this book includes a great deal of related information.

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Wool Research

The significance and present status of research on wool fiber. J. Nüsslein. *Reichsamt Wirtschaftsausbau* Prüf-Nr. **54** (PB 52006), 103–18 (1940) (through *Chem. Abstr.* **41**, 5311g (Aug. 20, 1947)).

A descriptive article containing photomicrographs and electron micrographs. The effects of a number of degreasing agents under varying conditions on treated woolens are shown in a series of bar graphs.

Text. Research J. Nov. 1947

Wool-Scouring Wastes

Wool-scouring wastes treated by new chemical process. Harry A. Faber and Paul F. Howard. *Water & Sewage Works* **93**, 467–73 (1946) (through *Chem. Abstr.* **41**, 5311*h* (Aug. 20, 1947)).

Scouring wool by means of alkali and soap produces a brown, turbid, putrescible emulsion of grease and organic matter containing on the average 12,000 p.p.m. of find solids in suspension. The previous methods of treatment—acid cracking and centrifuging—were only partially effective. The hypochlorite method described consists in mixing, aerating, then settling the waste with calcium hypochlorite approximately equal in amount to the alkali in the scour, to oxidize organic matter and permit separation of a sludge containing $CaCO_3$ and insoluble soaps; clear supernatant liquid is discharged. The sludge and scum are acidified with H_2SO_4 to pH 4–5, producing a concentrated sludge liquor, and a supernatant liquid which is discharged. Treated sludge, heated to 190°F, is filter-pressed, yielding a cake which dries readily, and a grease-water mixture, which is separated in a gravity separator, the grease concentrated, steamed, and acidified. Proceeds from the sale of wool grease exceed cost of operation. Hypochlorite process removes 98% of grease from water; 75% is recovered and sold. Plant installation at Abbot Worsted Co., Forge Village, Mass., is described.

Text. Research J. Nov. 1947

Textile Machinery

Standardization of textile machinery. A. Poncelet. *J. Text. Inst.* **38**, 225–34 (July 1947).

A broad review of the limiting factors concerning the standardization of worsted textile machinery. The author emphasizes the fact that animal fibers are a naturally highly variable raw material requiring fine classification and subdivisions into quality, place of origin, staple length, and other distinguishing characteristics. Because of these pronounced differences, the type of machinery and method of processing are to a very large extent instrumental in obtaining satisfactory processing results. Under these circumstances, no broad degree of standardization can be attained. However, among the suggestions given, one is for the standardization of textile machine design according to yarn quality and fineness. L. A. Fiori

Text. Research J. Nov. 1947

Textile Printing

Vat and azoic colors preferred for printing. A. K. Saville. *Textile World* **97**, 122–3, 190, 192, 195–6 (Aug. 1947).

A review.

Text. Research J. Nov. 1947

Report Writing

The essentials of a good report. Florence E. Wall. *J. Chem. Ed.* **24**, 129–40 (Mar. 1947).

This paper, presented before the Division of Chemical Education, American Chemical Society, Sept., 1946, was first prepared as part of a manual for the technical staff of a research laboratory. It briefly outlines reasons for writing reports, the types of reports issued by research laboratories, and the characteristics of a good report. Clearness, conciseness, and emphasis are the three qualities for which the author should strive. Details of the organization, writing, mechanical make-up, and processing of the report are given. The need for more courses in technical writing, rarely an innate skill, is emphasized.

Text. Research J. Nov. 1947 R. K. Worner

Byssinosis

Byssinosis in the cotton trade. C. I. C. Gill. *Brit. J. Ind. Med.* **4**, 48–55 (1947) (through *Chem. Abstr.* **41**, 4927*b* (Aug. 10, 1947)).

A review, with photographs, röntgenograms, and bibliography, of the respiratory disease affecting workers inhaling dust in cotton mills. The disease may be due to an irritating soluble protein in the cotton dust which penetrates into the alveolar tissue and gradually leads to thickening of the alveolar walls, or to superficial irritation of the bronchial mucosa by the dust, leading to chronic bronchitis, cough, and emphysema. An allergic basis is suggested.

Text. Research J. Nov. 1947

Occupational Eczema

Occupational eczema in the textile industry, ways and means of its prevention. Nils Hagström. *Färgeritekn.* **19**, 242–5; **20**, 5–6 (1944); *Chem. Zentr.* **1944**, II, 559–60 (through *Chem. Abstr.* **41**, 4926*i* (Aug. 10, 1947)).

The development of eczema is discussed and allergically sensitive cases are considered. Sensitivity tests carried out on workers in the dyeing room of a textile-finishing plant showed that 1% was the maximum NaOH concentration tolerated by the skin and that 0.5% solution at times caused eczema. Neither hypochlorite solution nor Naphthol AS alone was found to be irritating. Already coupled dyes were also without effect, whereas nitrosamines (Rapid Fast Orange RG and RH) caused serious irritation. Chloride of lime paste should be used for cleaning the hands only with the addition of soda and with subsequent washing with 10% $NaHSO_3$ and rinsing with water or dilute soap solution. It is recommended that the tables of functional skin tests reported by Heinze (cf. *C.A.* **33**, 6601[2]) be followed carefully.

Text. Research J. Nov. 1947